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AN INVESTIGATION OF DENTAL LUTING CEMENT SOLUBILITY AS A FUNCTION OF THE MARGINAL GAP

A

THESIS

Presented to the Faculty of
The University of Texas Graduate School of Biomedical Sciences
at San Antonio
In Partial Fulfilment
of the Requirements
for the Degree of
MASTER OF SCIENCE

By

Michael Steven Jacobs B.S., D.D.S.

San Antonio, Texas

May 1988

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DEDICATION

I would like to dedicate this thesis to my wife, Marilyn, for all her help and encouragement over the years.

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Special appreciation is extended to my Supervising Professor

Dr. A. Stewart Windeler

for his guidance, creative insight, and encouragement.

AN INVESTIGATION OF DENTAL LUTING CEMENT SOLUBILITY

AS A FUNCTION OF THE MARGINAL GAP

Michael Steven Jacobs, D.D.S.

The Universtiy of Texas Graduate School of Biomedical Sciences

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Dental cements used in fixed prosthodontics have the primary purpose of luting or sealing the cast restoration to the prepared tooth. A permanent luting cement should have high strength, low film thickness, be non-irritating to the dental pulp, and have low solubility. A critical property of luting cement is its solubility in oral fluids. If the cement dissolves at an unacceptably high rate the tooth is susceptible to recurrent caries and the retention of the cast restoration can be compromised.

It has been stated that the rate of luting cement solubility is related directly to the size of the marginal gap that exists between the cast restoration and the prepared tooth. (Cooper 1971; Johnston 1971) Thus, the larger the

marginal gap, the larger the resultant cement line and the more rapid the rate of cement dissolution. Fick's First Law of Diffusion, however, would predict that cement solubility as it relates to diffusion is dependent on the diffusion constant of the solute and concentration gradients . This would indicate that cement solubility as it relates to diffusion is independent of the mass of cement exposed. Presently, the exact relationship between the marginal gap size and the rate of cement dissolution is not known.

The purpose of this study was to investigate the rate of cement solubility as it relates to the degree of marginal opening. Both a static and dynamic environment was used to evaluate the influence of diffusion and convection on cement solubility.

Standardized test samples were made that would simulate marginal gaps and subsequent cement lines. The dimensions of these marginal gaps were 25, 50, 75, and 150 microns. Type 1 zinc phoshate cement was used as the luting agent. Phase 1 of the study evaluated test samples that were placed in a static solution. This allowed the investigator to study cement dissolution as it related to diffusion. Phase 2 of the study investigated the influence of a dynamic enviroment on the dissolution of cement.

At the end of the test periods, standardized photographs were made of the test samples. These photographs were used to measure and record the remaining areas of cement so as to compare the rate of cement dissolution as it relates to the degree of marginal opening.

A one-way ANOVA for the Phase 1 Diffusion Study revealed that there were statistically significant differences between the test groups. A Duncan's Multiple Comparison Test demonstrated that the 150 micron test group had a small but statistically significant decrease in the remaining dental luting

cement. The 25, 50, and 75 micron groups demonstrated no statistically significant differences in the amount of remaining luting cement. ($p < 0.05$)

A one-way ANOVA was also performed on Phase 2 Dynamic Study. This test revealed that there were statistically significant differences among the test groups. A Duncan's Multiple Comparison Test demonstated that there was a statistically significant decrease in the remaining cement in the 150 micron test group. The remaining cement in the 25, 50, and 75 micron test groups were not significantly different. ($p < 0.05$)

During the Phase 1 Diffusion Study two distinct regions of luting cement were visible in the test samples. A small outer "halo" of cement which was termed the "affected layer" surrounded a larger inner "unaffected core" of luting cement. These layers of cement were evaluated using X-ray diffraction techniques. The results of these studies indicated that zinc oxide was absent in the outer affected layer of luting cement while present in the inner unaffected core of cement. This suggests that zinc oxide is one of the first constituents of zinc phosphate cement lost due to dissolution.

The results of the Phase1 Diffusion Study tend to support Fick's First Law of Diffusion when the marginal gap is less than 75 microns. At the 150 microns level, however, there was a slight increase in cement dissolution which was statistically significant. The Phase 2 Dynamic study demonstrated that as the marginal gap increased there was a gradual increase in cement dissolution. This increase, however, was not statistically significant. At the 150 micron level there was a statistically significant increase in cement dissolution.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Page

LIST OF PLATES

I. INTRODUCTION

Dental luting cements in conjunction with the geometry of the tooth preparation provide the basis for the retention of the casting upon the tooth. Moreover, dental luting cements serve a vital function by sealing the small inevitable discrepancies which occur between the cast restoration and the prepared tooth.

Of the available dental luting cements, zinc phosphate cement has the longest clinical history. Pierce first described this cement in 1879. (Pierce 1879) Although this cement was initially to be used as a direct restorative material, it was rejected by the dental community because of its high rate of solubility. It was not until 1902 when Fleck described a zinc phosphate cement with improved physical properties that this cement became generally used. (Fleck 1902) Interestingly, the formulation of zinc phosphate luting cement which is used today is very similar to that described by Fleck.

For eighty years zinc phosphate luting cement has been widely used even though it has significant deficiencies. The most clinically relevant of these deficiencies is its solubility in oral fluids. (Phillips 1982) This dissolution may result in recurrent caries and may cause the loosening of the cast restoration. Because of their solubility, luting cements in general have been described as the "weak link" when restoring teeth with cast restorations. (Johnston 1971)

The rate of luting cement dissolution has been empirically related to the degree of marginal opening. (Cooper 1971; Johnston 1971) Thus, the larger the marginal gap and subsequent exposure of the dental luting cement to the

oral fluids, the more rapid the rate of cement dissolution. Fick's First Law of Diffusion, however, would predict that the rate of cement dissolution is independent of the degree of luting cement exposed. (Adams 1956) This Law also states that in a static environment the rate of cement dissolution is dependent upon concentration gradients and the diffusion constant of the cement solute. It should be noted that when the effects of abrasion are added, the rates of cement dissolution would not be expected to follow Fick's First Law of Diffusion.

The precise relationship between marginal gap size and cement dissolution is not known. (Smith 1982) The purpose of this study is to investigate the rate of zinc phosphate luting cement dissolution as it relates to the size of the marginal gap. Both a static and a dynamic environment will be employed to observe the influence of the marginal gap size on cement dissolution.

II. LITERATURE REVIEW

A. Historical Development of Luting Cement

Dental luting cement is traditionally made by incorporating a powder into a liquid. These components are mixed to a paste consistancy. The paste is then placed in the inner surface of the cast restoration and cemented onto the prepared tooth. After a short period of time, the paste sets to a hard mass and the excess is removed. Since cast restorations do not fit perfectly (Rosner 1963; Andrews and Hembree 1976), one purpose of the luting cement is to provide mechanical and/or chemical bonding between the cast restoration and the tooth. Moreover, it serves to seal and fill the small gaps which exists between the casting and the prepared tooth.

Historically, not all fixed restorations were cemented. In 1860, the pivot crown was composed of an ivory or natural tooth and the post was made of compressed hickory wood. The compressed wooden post was attached to the ivory tooth and the root which was to receive this crown was "cut to the margins of the gums and the canal was reamed to receive the post." (Wells 1901) The wooden post was shaped to fit the internal surface of the root canal and then placed into the prepared root. It was thought that the salvia of the mouth would cause the hickory wood post to swell and that this would hold the crown in position. This restoration had a very high tendency to loosen and was subject to recurrent abscesses.

Cement was first invented, in 1855, by Sorel when he combined zinc oxide with an aqueous solution of zinc chloride to form a cementitious mass. (Sorel 1855) In 1858, Feichtinger recommended that this cement be used in

dentistry. (Mellor 1929) One of the oldest dental luting cements, composed of zinc oxide powder and phosphoric acid, was introduced by Pierce in 1879. (Pierce 1879) This cement was initially lauded and predictions were made that it would be an outstanding filling material. Due to their solubiltiy, however, these initial dental cements soon proved to be clinically unacceptable. It was not until Fleck documented improvements in zinc phosphate cement that this cement was accepted by the dental community. (Fleck 1902)

The early practitioners of restorative dentistry recognized that a significant problem associated with luting cements was their high rate of solubility. In order to minimize this problem, Carmichel suggested that a minimal quantity of cement be utilized between the restoration and the tooth so that the metal and the tooth formed an almost inseparable union. (Carmichel 1901) Moreover, he raised the issue of marginal integrity and stated that "the edges of the fastener must be made tight enough to prevent cement dissolution." Lane, expressing his discouragement over how rapidly luting cements dissolved, stated that even the best fitting crowns have a cement line which would eventually dissolve and lead to the failure of the restoration. (Lane 1910) His slogan for the cementation of crowns was that one should "reduce the bulk of cement to the smallest possible minimum." Finally, Burgess emphasized that cement dissolution is inevitable and would lead to the ultimate failure of the restoration. (Burgess 1915) Thus, the restorative practitioners of the early 20th century recognized the problems associated with luting cement dissolution.

It is surprising to note that even with the improvements made in the past 100 years in dental techniques, equipment, and materials, only in the last twenty years has there been significant advances made in the area of dental

luting cements. (Smith 1982) Futhermore, zinc phosphate cement is used today in much the same manner and form as when Fleck described it in 1901.

B. Standardization of Dental Cements

The primary weakness of any cast restoration lies in the solubility of the cementing medium. (Johnston et. al. 1955; Phillips 1982) Therefore, one characteristic of an ideal luting cement would be for it to be insoluble in oral fluids. Currently, such a dental cement is not available. Since it was recognized that the physical properties of dental cements were less than ideal, certain standards were established in 1934 which would provide quality control for the commercially available dental cements. (Paffenbarger 1934)

These early standards for evaluating dental cement solubility were incorporated into the American Dental Association's Specification Test No. 8 for zinc phosphate dental cement solubility. (American Dental Association's Guide to Dental Materials 1978) As this test was initially employed, thin disks of zinc phosphate cement were prepared and weighted. One hour after their preparation, the disks were immersed into distilled water. After a seven day period, the disks were removed from the distilled water and the water was evaporated. The dry weight of the cement residue was compared to the weight of the original disks. The acceptable percentage of dissolution was one percent. (Paffenbarger et. al. 1934) Today, the specification test for zinc phosphate cement solubility still requires immersing thin disks of cement into distilled water one hour after their preparation. However, the disks remain in the distilled water for only twenty-three hours. Once again the specimens are removed from the water, the water is evaporated, and the amount of cement residue is calculated. The current acceptable maximum amount of solubility

and disintegration for zinc phosphate cement according to the American Dental Association Specification Test No. 8 is 0.2 percent by weight.

The clinical relevance of this test, however, has been questioned because the methods by which the cement solubility standards were established bear little resemblance to the way dental cements are actually used. (Beech 1983) First, distilled water is not an appropriate representation of the fluids of the mouth. (Wilson 1976) Moreover, a cemented casting exposes only a small cement line to the oral fluids. (Mesu 1982) Thus, the specification test does not conform to the ideal canon of material testing which is that a material should be tested in the mode in which it is used. (Wilson 1975) Since the deficiencies of this test were recognized, subsequent research followed which more clearly evaluated cement solubility. (Norman 1957; Wilson 1975; Richter 1975; Beech 1983)

C. In Vitro Research

Whereas the American Dental Association SpecificationTest No. 8 predicted low solubility for zinc phosphate cement, deterioration of the thin cement line surrounding the casting was common. Since initial in vitro research demonstrated that distilled water was not an adequate testing medium, Norman theorized that the failure of the luting cement was related to the dilute organic acids in the oral fluids. (Norman 1957, 1959) Therefore, he proceeded to investigate the rate of dissolution of zinc phosphate cements in a dilute acid environment. Cement samples were prepared and were suspended in test solutions of acetic, citric, and lactic acids. The specimens were removed from solution and the amount of residue was measured. With the exception of acetic acid, the zinc phosphate cement was considerably more soluble in the organic acids than the distilled water. These results suggested that there was the

potential for increased cement solubiltiy under oral conditions because the cemented restoration were constantly being bathed with fluids containing organic ions.

Norman investigated the solubility of zinc phospate and zinc oxide and eugenol cements. (Norman et. al. 1963) The results of this study indicated that zinc phosphate cement was more soluble in dilute acetic acid than zinc oxide and eugenol cement. Similarly, zinc oxide and eugenol cement was less soluble in water than the zinc phosphate cement. He also found that the sealing properties of the zinc oxide and eugenol cement was superior to the zinc phosphate cement.

Solubility of zinc phosphate cement has been examined as a function of time. (Eichner 1968) In this study, five specimens of eight different types of cement were made. These specimens were placed in weighing bottles and their rates of solubility were examined at 24, 48, 96, 192, and 382 hours respectively. The water that was used to evaluate the specimens was not changed. In the second part of the study, cement specimens were similarly prepared and examined, however, the water was changed on a daily basis. This study produced two interesting results. First, when the water was not changed, the rate of dissolution steadily decreased after the first 24 hours. Secondly, it was observed that dissolution occurred at a greater rate when the water was changed on a daily basis. The authors applied Fick's law of diffusion and concluded that dissolution is a process that consists of both diffusion and erosion. Ficks' law of diffusion states that the flux of solute atoms moving across a boundary decreases as the opposite side of the boundary becomes more saturated with solute atoms. Thus, when the water was changed on a daily basis, there was a flux of solute out of the cement. This increased solute

diffusion resulted in channel formations and cracks in the cement which in turn caused particles to break off. Hence, the increased diffusion of the solute lead to increased erosion of the cement.

The durability of zinc phosphate and silicate cements has also been examined. (Wilson, et. al. 1970) In this study, the authors addressed the discrepancies between the results of the specification test and in vivo clinical observations; particularly, the low rates of solubility predicted by the twenty-four hour specification test for zinc phosphate cement when compared with the silicate cements. In order to evaluate this discrepancy, the methodology required that test samples be placed in various test solutions of differing pH over prolonged periods of time. The authors concluded that the specification test was not suitable for comparing silicate and zinc phosphate cements because of the test's brevity and lack of an acidic environment. While evaluating zinc phosphate cement solubility, the authors noticed that during the first twenty-four hours the initial elute was phosphate. As the cement aged, however, zinc became the primary elute. The authors reasoned that the initial phosphate elute was related to the unreacted liquid in the cement and a soluble intermediate product. Whereas, after the first twenty-four hour period, the zinc phosphate cement had matured and the elute reflected a genuine erosion of zinc oxide particles from the cementing matrix and not a "washing-out" of product. The authors also observed that the amount of zinc and phosphate in the solution increased when the acidity of the test solutions was increased.

The early exposure of zinc phosphate cement to water was evaluated by Swartz in 1971. (Swartz 1971) His investigation demonstrated that zinc phosphate, hydrophosphate, silicophosphate, and red copper cements were all soluble when exposed to water within ten minutes after they were mixed. The

solubility of these cements decreased as the amount of time was increased between mixing the cements and immersing them into water. Thus, she recommended that cavity varnishes be placed on margins of cast restorations immediately after cementation. This early protection from contact with the oral fluids may enhance the resistance of the luting cement to dissolution.

In another study, an effort was made to incorporate fluoride into zinc phosphate cement so as to enhance caries resistance. (Freitas l973) Test samples were evaluated over a fifty-two week period. Although the authors found that the stannous flouride ion released from the cement enhanced the insolubility of the adjacent enamel, it was also noted that the solubility of the cement was increased. Hence, the authors concluded that further study was required.

Colorimetric analysis of the erosion products of zinc phosphate cement has been evaluated. (Wilson l974) The author suggested that since phosphate is the matrix forming anion, a measurement of the phosphate anion would provide an indicator of cement solubility. He found that phosphate became progressively more fixed as the reaction between zinc oxide and phosphoric acid continued. Zinc, magnesium, sodium and intermediate matrix by-products were also eluted. The author stated that this simple colorimetric test could replace the gravimetric standardized test used to evaluate cement solubility.

The significance and validity of the specification test as it relates to cement solubility has been questioned. (Wilson l976) This author indicated that one deficiency of the specification test is that a clear physical measurement based upon chemical composition is not addressed. Instead the specification test expresses solubility and disintegration as a percentage of the weight of the

original specimen. Luting cements, however, are not simple substances and the dried residue which is used to calculate solubility should be identified according to composition. Moreover, since the specification test is of such a short duration, it is difficult to reach any conclusions as to the nature of the solute which is initially leached from the cement. For example, when luting cements are initially mixed there are reaction intermediate products which are very soluble. Also, the matrix of this recently mixed cement is soluble. However, as zinc phosphate cement continues to harden, it reaches a stage where the cement is simply zinc oxide particles embedded in a zinc orthophosphate matrix. This mature cement has a constant and low rate of dissolution. Thus, the specification test measures the solubility of an initially mixed cement; and, it does not provide an accurate prediction of the long term solubility of this cement.

In other studies cement dissolution was made visible. (Mesu 1982) The author of this study recognized that luting cements are only exposed to the oral fluids as very thin cements lines. Therefore, in this study, a luting cement was mixed and placed between clear, circular quartz disks which were designed to simulate the dimension of a clinical margin. The disks were initially photographed, placed in test solutions, and photographed at various times during the study. The photographs were used to compare the various rates of dissolution between the samples and could also be compared with the original photographs to determine the area of dissolution. This was accomplished by measuring the undissolved areas directly from the photographs. Mesu described the following stages of degradation: solution, disintegration, absorption, and the unattacked core. Thus, a unique aspect of this study was the visibility of the dissolution. The author also attempted to evaluate cement

dissolution as it related to size of a marginal gap. He placed a 100 micron wire between the quartz disks and attempted to establish a marginal gap of 100 microns on one side of the disk and a twenty micron marginal gap on the opposite side. For standarization reasons, however, further experiments were restricted to the plane parallel placement of glass plates.

New methods have been employed for evaluating in vitro cement solubility. (Beech and Bandyopadhyay 1983) Beech and Bandyopadhyay stated that the use of the specification test for cement solubility of different types of cements was meaningless and potentially misleading. In order to make the in vitro testing of cement solubility clinically relevant, they developed a test for dissolving test cements which employed jets of fluid. The authors, recognizing that low pH conditions are experienced in the mouth, employed eroding fluids of various pH's to dissolve their test samples. The authors stated that since margins are generally located in stagnant areas, the cement lines of luting cements may be subject to a pH less than seven. They indicated that lactic acid would be a prime product of bacterial plaque in these areas. An advantage of their testing system was that it produced quick reproducible values of cement dissolution. It should be noted that these values correspond to in vivo studies of cement solubility.

Walls also developed a new erosion test for dental cements. (Walls 1985) Erosion was defined as the extent to which a material is degraded by its environment. The author stated that intraorally the luting cement residue was constantly being washed away so as to expose new unaffected cement. The purpose of his study was to mimic this phenomena and provide a more clinically relevant test. Cement test samples were fabricated and periodically raised and lowered into a test solution. At the end of the test cycle, a profilometer was used

to measure cement dissolution. Walls ranked the five investigated cements in the order of their solubility: KETAC FIL (least soluble), SILICAP, ZINC PHOSPHATE , ASPA, and DURELON (most soluble).

While it is realized that duplication of the exact conditions of the mouth is difficult, if not impossible, the aforementioned authors attempted to investigate luting cements in a manner which reproduced the environment in which these cements are used. Thus, the common theme running throughout these studies was the authors' attempt to make the testing of cement solubility more clinically relevant.

D. In Vivo Research

The initial clinical studies involving luting cement solubility were reported by Norman. (Norman 1969) He used specially constructed removable partial dentures to test cement solubility. A lingual window was cut on the metal framework of the partial denture; thus, an in vivo study of cement solubility was possible while the partial denture was worn by the patient. The partial dentures were weighed and the cement was inserted into the windows. The partial dentures were then placed in a humidor for twenty-four hours. At the end of twenty-four hours the partial dentures were wiped dry and again weighed. The partial dentures were given to eight patients with instructions that they were to wear the partial denture continuously for a thirty day test period. The cements which were investigated included zinc phosphate, silicate, and zinc oxide eugenol. The results demonstrated that silicate cement was less soluble than zinc phosphate and that zinc phosphate was less soluble than zinc oxide eugenol cement. One possible flaw in this study was the location of the cement window. Because the window was placed on the lingual surface of the partial denture, it was subjected to high levels of abrasion from the tongue. Moreover,

the area of the cement window was much greater than would normally be exposed in a clinical situation.

Norman's study motivated Richter to evaluate the solubility of dental cements in areas not subject to abrasion. (Richter 1975) This study involved nine patients each of whom required mandibular fixed partial dentures. Receptacles which were 3mm in diameter and 2mm in depth were cut in the pontic area. Two receptacles were placed on the facial surface of the pontic, two were placed on the lingual surface, and four were placed on the pontics ridge lap. Cement was then placed in these receptacles. Silicon impressions were made on the pontic area and the surfaces of the eight cement specimens. Silver plated models were made from these impressions and the models were photographed. The fixed partial dentures were cemented in the patients' mouth with a temporary cement. After one year, the fixed partial dentures were removed and new impressions were made of the pontic area. New silver plated models were made and photographed. The initial photographs were then compared with the subsequent photographs so as to evaluate the amount of dissolution of cement. Richter's results confirmed Norman's inference that the areas subject to abrasion, such as the facial and lingual surfaces, had a greater degree of cement degradation than areas not subject to abrasion. Even though the ridge lap area of the pontic was not subject to abrasion, a considerable loss of cement was recognized. This loss, however, was less than that which occurred in the facial and lingual areas.

Another in vivo study of the solubility of dental cement was conducted using a cement sample holder containing six different dental cements placed in a slot which was prepared in a complete maxillary denture. (Mitchem 1978) The sample holder was then covered with a perforated plastic cover which would

permit the oral fluids to bathe the cement samples but would prevent the effects of abrasion. The dentures were placed in the patient's mouth. After six months the sample holder was removed from the denture and the amount of cement dissolution was measured. The glass ionomer cements demonstrated the least amount of solubility, followed by zinc phosphate and silicophosphate cements, respectively. The author, emphasizing that his study eliminated the abrasion factor, concluded that his results provided strong evidence of which cements were the most and least soluble in the oral fluids.

A similar in vivo study was conducted using complete gold crowns as the cement carrier. (Osborne 1978) Four holes 0.82 mm in diameter and 1.23 mm in depth were placed on the interproximal surfaces of the gold crowns. Two holes were located near the gingival margin and two near the occlusal so as to simulate a gingival and occlusal margin. Each of the holes were filled with a different cement. The crowns were cemented in the patient's mouth for six months. After the test period the crowns were removed and the amount of cement dissolution was evaluated. A dial guage was used to directly measure the loss of the dental cement. The author stated that the two advantages of this procedure were that the effects of abrasion were minimized and that the location of the cement approximated clinical usage. Interestingly, Osborne conducted an in vitro test utilizing the same cements as used in his in vivo test. There was no correlation between the results of the in vitro and in vivo tests for screening cement solubility.

Mesu conducted an in vivo study to correlate its results with his previous in vitro findings regarding cement solubility. (Mesu 1983) Using specially prepared parallel plate glass test samples, cements were mixed and placed between the plates. These samples were then placed in the second molar

region of a mandibular complete denture. The dentures were then worn by the patients for a prescribed test period. Mesu then compared his in vivo results with the in vitro results for samples placed in an artificial salvia and a 0.02 mol (pH 4) solution of lactic acid. Although there was no correlation in cement solubility between the in vivo test results and the artificial saliva, the lactic acid solution demonstrated a strong correlation. Thus, to predict in vivo cement solubility the 0.02 mol (pH 4) lactic acid provides a good in vitro test solution.

In another in vivo study of cement solubility, cement samples were placed into holes which were cut in specially prepared bovine incisors. (Pluim 1984) The cements which were investigated included zinc phosphate (Standard) and a glass ionomer cement (Chem Bond). The incisors were incorporated into the buccal flange area of a mandibular complete denture. The denture was worn for a period of six months. At specified intervals, replicas of the cement surface were made and quantified using SEM stereographic photographs. The results of this study demonstrated in vivo solubility rates of 80 microns per week for zinc phosphate and two microns per week for glass omer cement.

Theuniers's investigation confirmed Pluims' findings that glass ionomer cements were less soluble than either polycarboxylate cement or zinc phosphate. (Theuniers 1984) The test samples consisted of enamel cylinders which were calibrated to created cement film thicknesses of 25 to 100 microns. The samples were then cemented into metal containers. In the in vivo phase of this study, the test samples were mounted in a telescopic fixed partial denture which were then cemented into the patient's mouth. In the in vitro phase of the study, similar test samples were placed into water, acetic acid, and enzymes; and, at the end of the test period the cement solubility was evaluated. It was

determined that glass ionomer cements were the least soluble of the cements tested. Moreover, acetic acid was the only test solution which approached the destructive capability of the oral environment.

Wolff investigated cement film thickness as it related to cement solubility. (Wolff et. al. 1985) In this study, four countersink holes with radii of 25, 50, 100, and 200 microns were milled into cast crowns. A 1 mm precision machined gold pin was fitted into the center of the hole creating a gap of 25, 50, 100, and 200 microns between the pin and the casting. The gold pin was then cemented with zinc phosphate cement thereby creating a cement line of the aforementioned dimensions. A total of six castings were fabricated and cemented in the patient's mouth. After six months the crowns were removed and the degree of cement dissolution was measured. It was found that minimal cement loss of 20 microns; and contrary to empirical opinion, there was no discrepancy in cement solubility between the 25 micron and 100 micron cement margins.

In another investigation, hollow orthodontic tubing and the acid etch composite resin technique were used to evaluate cement solubility. (Ibbetson 1985) Hollow orthodontic tubing, with a 0.05 mm diameter, was filled with dental cement and attached to the maxillary first molar using the acid etched composite technique. The tubing remained in the patient's mouth for 90 days. The tubing was removed and the amount of cement dissolution was measured with a microscope. The study indicated that glass ionomer cements were less soluble than either zinc phosphate or polycarboxylate cement.

Finally, the relationship between salivary properties and in vivo cement solubility was investigated. (Pluim and Arands 1987) This study evaluated the effect of saliva's buffer capacity and pH upon the in vivo solubility of two zinc phosphate and two polycarboxylate cements. Test samples containing the

cements were inserted into the mandibular dentures of ten patients. The dentures were given to the patients with instructions that they were to wear the dentures continuously; and, that the dentures could be cleaned but not with commercial cleansers. Cement loss was evaluated at 1, 2, 3, 5, 8, 12, and 24 week intervals; and, one-half of the samples were evaluated at the end of 48 weeks. The amount of cement loss was determined by stereoscopic SEM measurements on replicas of the cement surface. Moreover, the saliva of each participant was characterized for its buffering capacity and its pH. The results indicated that there was no relationship between in vivo solubility and the pH or buffering capacity of the saliva. The author concluded that dental luting cement solubility is caused by the acids from dental plaque and food but not the saliva itself.

E. Chemical Properties of Zinc Phosphate Cement

It has been stated that no material used by the dentists involves more applied chemistry than dental cements; therefore, a knowledge of the chemistry of dental cements is important in the proper manipulation and use of these products. (Crowell 1927) All dental cements are based upon a hardening reaction between a powdered solid and a visceous hydrogen-bonded liquid. The liquid acts as a hydrogen proton donor while the powder, a slightly basic substance, acts as the proton acceptor during the cement formation reaction. (Wilson 1978) Therefore, dental cements may be described as acid-base reaction cements. Moreover, as the cement is formed, hydrogen bridges in the liquid phase are replaced by more rigid metal ion bridges. (Wilson 1978) This causes the liquid to gel and the gel to harden. Finally, it has been noted that the matrix of all dental cements are amorphous. (Wilson 1978) For example, mature

zinc phosphate cement consists of zinc oxide particles embedded in an amorphous cement matrix.

The oldest and simplest of the permanent luting cements is zinc phosphate. When this cement is first mixed it has a low initial pH, then an exothermic reaction occurs. (Craig 1985) Within three minutes after the start of the mix, the zinc phosphate cement will reach a pH of 4.2 and will rise to a pH of 6 within one hour. After forty-eight hours the pH of the cement will be neutral. Despite possible injury to the tooth due to the exothermic reaction and low initial pH, zinc phosphate cement is still the most widely used class of dental luting cements. (Smith 1976; Abelson, 1980)

The composition of today's zinc phosphate cement is similar to that used fifty years ago. (Smith 1982) The powder, composed primarily of zinc oxide and 2-10 percent magnesium oxide, has been sintered at over 1000 degrees centigrade for several hours. Both the firing of this powder and the magnesium oxide reduces the reactivity of the zinc phosphate cement and extends its working time. The liquid phase of this cement is composed of 45-64 percent phosphoric acid, 30-55 percent water, 2-2.5 percent aluminum, and 1-9 percent zinc. The function of the aluminum is to aid in the formation of the glassy zinc phosphate matrix, while the zinc serves to slow the reaction between the powder and the liquid.

The set zinc phosphate cement is a mixture of unreacted powder particles embedded in a matrix of amorphous zinc alumino-phosphate gel. (Smith 1982) This matrix, however, is not completely stable. Thus, when the surface of the zinc phosphate cement is exposed to water crystallite of hopeite develops at its surface. Such crystallization destroys the bonds which exists

between the cement and theprepared tooth. For this reason, it is important to avoid liquid contamination following cementation. (Swartz 1971)

The structure of zinc phosphate cement has been evaluated using X-ray diffraction techniques. (Servais and Cartz 1971) The authors investigated the sensitivity of the amorphous non-crystalline phosphate matrix of zinc phosphate cement when exposed to high humidity and water. To evaluate this phenomena four groups of test samples were created: Group A--the cement was mixed and stored in 30 percent relative humidity; Group B--cement was mixed and placed in 100 percent relative humidity; Group C--cement was mixed and immersed in distilled water; and Group D--cement specimens were mixed and placed between two glass plates. X-ray diffraction studies were conducted on all four groups. The results revealed that the hopeite was found only in Groups B, C, and D. While the hopeite formation was anticipated in Groups B and C, the authors explained that the formation of hopeite in Group D was the result of excess moisture being trapped between the glass plates. Moreover, the enclosure of cement between the glass plates simulated what occurred when cement was placed in a cast restoration and onto a tooth. In both instances, the enclosed cement was subject to excess moisture because of the lack of evaporation. Thus, the formation of the weakly bonded hopeite provides one additional explanation for this cement's lack of adhesive property.

In a follow-up study, zinc phosphate cement was placed between a glass plate and a prepared slice of tooth structure. (Cartz et. al. 1972) This study confirmed earlier results because once again crystallization occurred when the zinc phosphate cement was placed in an enclosed space.

F. Physical Properties of Zinc Phosphate Cement

The physical properties of zinc phosphate cement which are important for clinical success are strength, film thickness, and solubility. (Craig 1985) The American Dental Association Specification Test No. 8 has addressed each of these properties. First, the minimum compressive strength of zinc phosphate cement after twenty-four hours is 75 MN / sq. mm or 10,900 lbs per sq. inch. Second, the maximum film thickness for Type I zinc phosphate cement is 25 microns. Finally, the maximum solubility in water after twenty-four hours is 0.2 percent.

Long-term clinical success using zinc phosphate as a luting cement has been documented, but its physical properties, while adequate, are not ideal. (Farah 1985) Jorgensen presented a study which evaluated the crushing stength of zinc phosphate cement as it related to a cast restoration. (Jorgensen 1967) This study revealed that as the crushing strength of the zinc phosphate cement increased so did its retentive capabilities. The study also demonstrated that the crushing strength increased as the powder-liquid ratio increased.

In another study, the crushing strength of zinc phosphate cement was compared to other types of luting cements. (Powers 1976) It was found that zinc phosphate had the highest values of compressive strength when compared to zinc polycarboxylate cement and zinc oxide and eugenol cement.

The stress / strain behavior of zinc phosphate has also been evaluated. (Oilo and Espevik 1978) These investigators fabricated cement cylinders which were compressed until they fractured. The results indicated that zinc phosphate cement exhibited high strength, high modulus of elasticity, and a small plastic strain at fracture. The values of zinc phosphate cement were significantly higher than polycarboxylate or EBA cement.

Mixing zinc phosphate on a cold glass slab has been investigated. (Jenderson 1973) When a glass mixing slab was chilled to 7 degrees centigrade it was demonstrated that 26-46 percent more powder could be incorporated in the liquid, the working time was doubled, and the setting time decreased 30-57 percent. It was also noted that the compressive strength, solubility, and tensile strength of zinc phosphate cement when mixed on a chilled slab was comparable to a standard mix of zinc phosphate cement.

In an attempt to provide more working time and to possibly enhance the physical properties of zinc phosphate cement, the cement was mixed on a frozen glass slab. (Tuenge 1978) This study revealed that the compressive strength of zinc phosphate mixed on a frozen glass slab satisfied the standards of the American Dental Association's Specification Test No. 8. By utilizing the frozen slab technique, more powder could be incorporated into the same volume of liquid and this would offset the effects of water condensation on the frozen slab.

The minimum allowable film thickness for Type I zinc phosphate is twenty-five microns. Several factors which influence the final film thickness of the luting cement include grain size, cementation pressure, powder-liquid ratio, convergence angle of the preparation, and occlusal venting. (Jorgensen 1960; Eames 1978) Jorgensen evaluated the aforementioned factors and found that cement film thickness of approximately twenty microns could be produced when all of the other factors were optimal.

Changes in the film thickness of zinc phosphate cement has been used as a measure of its working time. (Windeler 1978) In this study sequential measurements of the film thickness of zinc phosphate cement permitted the working time of this cement to be quantified. The author noted that initial

increases in film thickness marked the end of the working time. Moreover, this study also demonstrated that: (1) a reduction in mixing temperature significantly increased working time, (2) the addition of a small amount of powder to the liquid a few minutes before mixing did not extend working time, and (3) incorporating small amounts of water into the cement mix decreased setting time but did not affect working time.

The influence on film thickness of a powder enriched mix of zinc phosphate cement and a decreased mixing temperature has been evaluated. (Windeler 1979) Standard mixes of Fleck's, S.S. Whites, and Ames zinc phosphate cement were made and the film thickness of these cements were measured utilizing a modified American Dental Association's Specification Test No. 8 and a tapered die system. All of these cements demonstrated a decrease in film thickness when the temperature of the mixing slab was reduced. When the modified American Dental Association's Specification Test No. 8 was utilized, only the Ames cement demonstrated a statistically significant reduction in film thickness when an increased powder to liquid ratio was tested under reduced mixing temperatures. Both the Fleck's and the S.S. White cements, however, demonstrated small but not statistically significant decreases in film thickness. When the mixing temperature was reduced and the powder enrichment was evaluated using the tapered die system, Ames cement showed an insignificant reduction in film thickness whereas both Fleck's and S.S. Whites demonstrated an increased film thickness. The increase in film thickness when using the tapered die system illustrates the need for appropriate relief on the internal surfaces of casting. This relief should be equal to the film thickness of the cement in order to enhance seating to the cast restoration.

The influence of grain size on the film thickness of zinc phosphate cement was also described. (Jorgensen 1963) The author stated that the grain size of the residual powder was one of the determining factors in the ultimate film thickness of the cement. Moreover, it was noted that the seating accuracy of the precision restoration was enhanced by utilizing a fine grain cement.

Finally, the hydrodynamic conditions which exist during cementation were also investigated. (Jorgensen 1960) In this article, Jorgensen described the effects of filtration. Filtration occurs when lumps of zinc phosphate cement become trapped by the narrowing space between the cast restoration and the prepared tooth. The lumps of powder act as a filter separating the cement particles from the liquid. The net effect of filtration may be that it leaves areas under the crown almost completely powderless, it decreases the retention of the crown, and pulpal injuries may result from the accumulating phosphoric acid. The effects of this hydrodynamic phenonema can be essentially eliminated by placing an occlusal vent in the crown.

Phillips has stated that cement solubility is the most important physical factor when evaluating zinc phosphate cements. (Phillips 1982) Since this subject was previously discussed, I would refer the reader to the earlier in vivo and in vitro discussions.

G. Margins

1. Marginal Fit

The typical cemented cast restoration presents a thin cement line the size of which is directly related to the marginal opening. A common goal when fitting and finishing a cast restoration is to minimize the inevitable cement line. While various authors have empirically related the rate of cement solubility to the degree of marginal opening (Cooper 1971; Johnston 1971), at present, the

influence of the marginal opening on the rate of cement solubility is not known. (Smith l982)

Recent studies has demonstrated that castings which were cemented with no accomodations for cement film thichkness could fail to seat by as much as l00 microns. (Fusayama l964; Cooper l971; Eames l978) In order to minimize the exposure of cement various types of marginal configurations have been developed and evaluated. (Rosner 1963; Fusayama 1964; Gavelis 1981; Pascoe 1983)

Studies have been conducted to evaluate the marginal fit of castings. (Christensen l966) The author evaluated the ability of ten experienced dentists to detect marginal discrepancies and determine which discrepancies were clinically acceptable. The mean acceptable opening in the gingival area was 74 microns (range of 34-ll9 microns). In areas of visible margins, the least acceptable margin was 34 microns (range of 2-51 microns). The author concluded that the evaluation of visibly accessible margins was more consistant and reliable than the evaluation of gingival margins. Also, subgingival margins which were deemed clinically acceptable would probably have been rejected as unacceptable if the clinician had been able to visually evaluate them.

In a similar investigation, Dedmon fabricated an instrument that would simulate marginal gap discrepancies. (Dedmon l982) The author selected six experienced clinicians to evaluate these marginal gaps. The blindfolded subjects were given an explorer and were asked to rate the marginal gaps using only their tactile senses. The acceptable non-visible marginal gaps had a mean equal to 104 microns (range of 32-230 microns). This study demonstrated that the acceptable mean for vertical margins was ll4 microns verses 93 microns for the horizontal margins. The author concluded that when

evaluating non-visible margins with an explorer, acceptance was more likely to be based upon the size of the overhangs and ledges than on the actual size of the marginal opening.

The effects of limited physical access to non-visible margins was investigated by the same author in a following study. (Dedmon 1985) Once again, six experienced clinicians were asked to evaluate marginal discrepancies created by a specialized instrument. The results indicated that the acceptable mean marginal opening was 53 microns. If one were to add an additional 20 microns to simulate the effects of cementation, the resulting 73 microns cement line duplicated Christensen's results. The author concluded that the marginal discrepancies which resulted from incomplete seating were difficult to detect; and, that it is not unreasonable to assume that larger than optimal marginal discrepancies may be deemed clinically acceptable.

McLean evaluated cement film thickness by using an in vitro technique. (McLean 1971) A polyether rubber impression material was placed inside the cast restoration and cemented upon the prepared tooth. The cast restoration was removed from the tooth after the polyether material had reached its final set and had adhered to the cast restoration. The casting was then filled with a mix of an acrylic resin. After the resin reached its final set, the cast restoration was removed from the rubber film and the resin core. The rubber film which was embedded on the acrylic resin was sectioned, and the film thickness for all the restorations was examined. The mean film thickness for the axial walls was 92.9 microns, while that of the occlusal floor was 112.3 microns. Therefore, McLean concluded that the term "goo clinical fit" may have several interpretations. Moreover, he suggested that marginal discrepancies in the order of 40 to 80 microns would be difficult to detect.

In a longitudinal study, McLean evaluated the marginal discrepancies of over l000 cast restorations. (McLean 1971) This study indicated that clinical success was possible when cast restorations exhibited marginal gaps in the range of 10 to 160 microns. Moreover, the author suggested that a successful cast restoration was possible if the marginal gaps and subsequent cement film thickness was less than 120 microns.

2. Marginal Design

To minimize the cement line various types of marginal configurations have been advocated. (Rosner l963; Pascoe l983) Rosner advocated the placement of a bevelled margin. He stated that the bevelled margin reduced the inherent defects in the casting and cementation procedure because it protects the enamel rods, allows for burnishing, and develops circumferential retention. While Pascoe concurred that a bevelled margin would reduce marginal discrepancy, he maintained that a shoulder margin provided better overall seating of the cast restoration.

Gavelis evaluated marginal configurations to determine the relationship between the marginal seal and the restoration's degree of seating. (Gavelis 1981) The author demonstrated that the feather-edge marginal design produced a marginal seal of 31-34 microns; the parallel bevel preparation produced a marginal seal of 41-44 microns; the shoulder preparation produced a marginal seal of 67 microns; and the 45 degree shoulder with a 30 degree bevel produced marginal spaces from 95-l05 microns.

3. Marginal Materials

The use of fixed prosthodontics as a treatment modality has increased during the past thirty years. Moreover, during this time, there has been a gradual shift from the use of precious metals to nonprecious metals for the

fabrication of fixed prosthesis. This movement was accelerated in 1968 when the United States Government discontinued their maintanence of the price of gold at thirty-five dollars an ounce. Since 1972, the price of gold has increased tenfold and has fluctuated at these high levels. Thus, economics has been one motivating factor in the shift from precious to nonprecious metals.

In 1976, approximately twenty-five percent of all crowns and fixed partial dentures in the United States were fabricated with nonprecious metals. (O'Brien 1977) Moreover, a 1981 survey indicated that seventy percent of the dental labs in the United States were used non-noble alloys to some degree. (Haskell 1982) Although the new base metals have superior mechanical properties when compared with the precious metal (Moffa 1973), the ability of these alloys to cast accurately has been questioned by several authors. (Nitkin 1976; Vincent 1977; Duncan 1982)

Nitkin fabricated two complete crowns and two three-quarter crowns using eight different nonprecious and precious dental alloys. (Nitkin 1976) Ten judges were selected to evaluate the fit of the test castings. The results indicated that the nonprecious alloys were inferior in fit and that they had larger marginal descrepancies than crowns cast with precious metal alloys.

The type of alloy which provided the best marginal fit for porcelain-fused-to-metal restorations was investigated. (Vincent l977) The study revealed that precious metals demonstrated a superior ability to cast test samples when compared to nonprecious metals.

Finally, the marginal integrity of precious metal castings were compared with the marginal fit of nickel-chromium alloys. (Duncan l982) Ten crowns each of a precious metal and four nickel-chromium alloys were fabricated on a test die and the subsequent fit of the crowns was measured. The difference

between the prepared margin and the casting margin has been termed "casting discrepancy." The mean casting discrepancy for the precious alloy was 111 microns. The mean discrepancy of the four nickel-chromium alloys were respectively 237 microns, 373 microns, 451 microns, and 463 microns. The author concluded that nickel-chromium alloys produced a greater casting discrepancy than did the precious alloy.

H. Periodontal Considerations

In fixed prosthodontics some degree of marginal discrepancy will exist, therefore, a small cement line is inevitable after the casting is cemented. If these marginal discrepancies are subgingival, the potential for gingival inflammation increases. (Loe 1968; Silness 1970; Romanelli 1980; Wilson 1981) This is one reason why the placement of supra-gingival margins has been advocated. (Grosso and Carreno 1978; Tjan 1980; Becker and Kaldahl 1981; Behrano 1982; Schillingburg 1982) However, the clinician may be forced to place the preparation's margin subgingivally due to subgingival caries, previous subgingival restorations, or the need to increase the resistance and retention form of the preparation. When this occurs, the rough surface of the luting cement will be directly exposed to the intercrevicular tissues and fluids; and, the potential for plaque accumulation and subsequent gingivitis and periodontitis may be enhanced. (Waerhaug 1953; Waerhaug 1960; Palamo and Pedens 1976)

The marginal fit and its relationship to periodontal bone levels has also been evaluated. (Bjorn 1976) In this study, 225 sets of 22 intraoral film x-rays were used to evaluate marginal fit. The study revealed that 83 percent of the gold castings and 74 percent of the porcelain restorations had deficient margins.

The area of the exposed cement has been calculated on the basis of marginal design and discrepency. (Silness 1970) The author evaluated the area of exposed cement for several marginal designs including the chisel edge, shoulder, and shoulder with a bevel. The study revealed that doubling the convergence angle of the chisel edge marginal design, the amount of cement exposed was doubled; and, when the marginal discrepency was doubled the area of cement exposure was proportionately increased. It was also determined that a direct relationship existed between the marginal discrepency and the area of exposed cement for the shoulder preparation. For example, a 0.2 mm marginal discrepency would produce a total area of 6.28 mm of exposed cement; and, a 0.3 mm marginal discrepency would yeild a 9.4 mm area of exposed cement. Thus, it was concluded that the shoulder preparation produced a greater exposure of cement than the chisel edge marginal design. It was also noted that the bevelled shoulder produced a smaller area of exposed cement than the shoulder; and, that as the bevel angle paralleled the axial surface of the preparation, the percentage of exposed cement significantly decreased. As a result of these findings, the author recommended that a bevel shoulder be used to minimize the cement line. Moreover, he emphasized the disadvantage of introducing several square millimeters of rough luting cement surface to the oral environment. Since this surface will frequently be covered by plaque, every effort should be made to minimize this cement- tissue interface by the supragingival placement of margins.

III. METHODS AND MATERIALS

A. Fabrication of the Twenty-five Micron Control Test Samples

Although various authors have studied cement solubility, only Mesu and Wolff have investigated the solubility of dental luting cements in the dimensions in which they are actually presented to the oral environment. (Mesu 1982; Wolff 1985) Interestingly, the results of these two authors conflicted with regard to zinc phosphate solubility. Since dental luting cements are generally presented to the oral fluids as a thin cement line, it would seem logical to evaluate cement dissolution in the same physical dimensions that are found clinically. In order to reproduce these dimensions, special test samples were fabricated.

Each test sample consisted of a thin measured layer of zinc phosphate cement interposed between two transparent quartz disks. To fabricate the test samples, seventy standard sized circular quartz disks with plane-parallal flat surfaces were purchased. (Quartz Scientific, Inc., 819 East Street, Fairport Harbor, Ohio 44077) (Plate 1) These disks were measured with a micrometer to insure that they satisfied the required specifications, i.e. 25.4 mm in diameter and 6 mm thick. The disks were meticulously cleaned in an ultrasonic cleaner, washed in distilled water, and dried. The disks were then paried by placing them on top of one another and an initial baseline measurement was recorded of the combined thickness. (Plate 2) To insure measurement in the same locations both before and after cementation, the investigator utilized three different color pens to place vertical lines equal distance from one another on the external edges of the paired disks. The four baseline measurements were

Plate 1. Paired quartz disks.

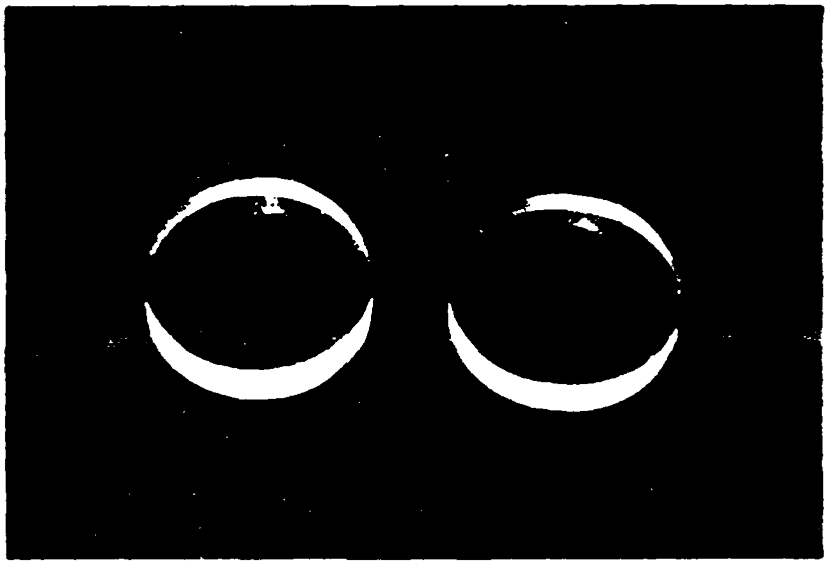
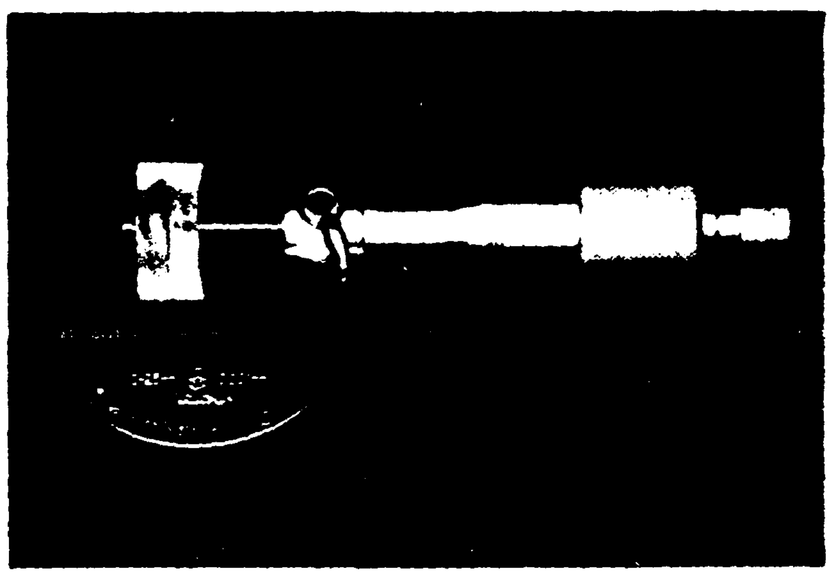


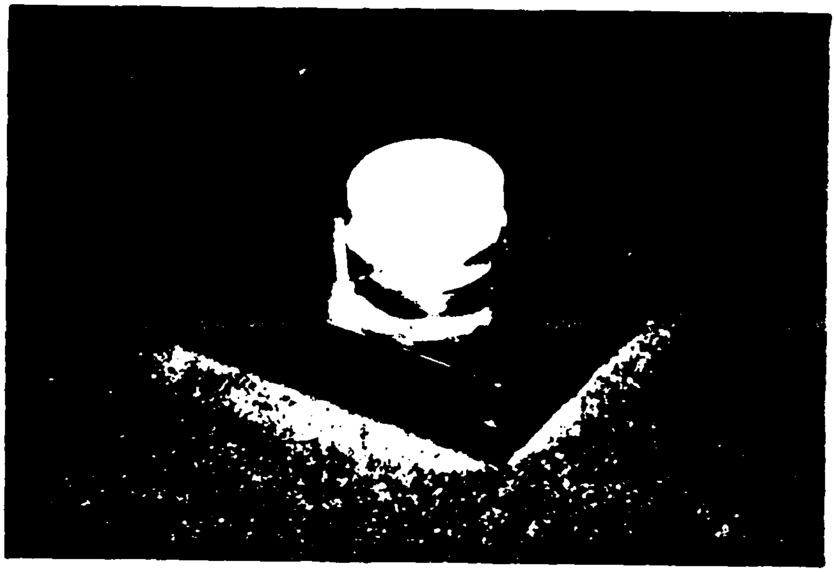
Plate 2. Initial pre-cementation baseline measurement of the combined thickness of the quartz disks.



made using a micrometer. These measurements were at the reference lines and the center of the paired disks. The measurements were recorded and then averaged. The mean of the four measurements represented the pre-cementation thickness of the paired disks.

The luting cement which was selected for use in this study was zinc phosphate cement. (Fleck's , Mizzy, Inc. Clifton Forge, Virginia 24422) The mix of the luting cement was standardized by using 1.6 grams of zinc phosphate powder and 0.6 ml of phosphoric acid liquid for each test sample. The powder was weighed and the liquid was measured with a 1.0 ml tuberculin syringe. All of the powder and liquid which was used to create the test samples came from the same lot number of zinc phosphate cement; and, this investigator mixed the cement for each individual test sample. The following mixing procedure was utilized which was in accordance with the American Dental Association's Specification Test No. 8 : (1) a clean glass slab and cement spatula were prepared for mixing the zinc phosphate cement, (2) the weighed powder was placed on the right half of the glass slab and separated into six equal sections, (3) the liquid was placed upon the left half of the glass slab, (4) a stop watch was started when mixing began, (5) the cement was mixed over a wide area of the glass slab and each of the six sections of powder were incorporated at incremental l5 second intervals, and (6) the mixing was complete at the end of 90 seconds. The mixed zinc phosphate cement was then placed between the two quartz disks and the disks were realigned according to the reference lines. The cemented quartz disks were placed in a specially fabricated alignment fixture which was designed to insured that the edges of the quartz disks were in the same vertical plane. (Plate 3) Once aligned, the quartz disks were placed and centered under the vertical arm of a precision press and a load of l4.7

Plate 3. The cemented quartz disks positioned in the alignment fixture.



Newtons (15 kilograms) was applied 120 seconds after the start of the mix. (Plate 4) Any excess zinc phosphate cement which extruded from the paired disks due to the load was carefully removed before the initial set of the cement. Fifteen minutes after the start of the mix, the quartz disks were removed from the precision press and examined to insure the continued alignment of the reference lines. After cementation, the four reference areas were remeasured using the same micrometer. The thickness of the cement interspersed between the quartz disks could be calculated by subtracting the initial measurement of the disks from the post-cementation measurement. This increase in the post-cementation measurement corresponded to the minimal film thickness of 25 microns for Type 1 zinc phosphate cement. Next, 600 grit sandpaper was used to create a flush external cement line which was in the same vertical plane as the edges of both quartz disks. A stainless steel clamp, which was engraved for identification purposes, was placed around the test sample so as to secure the cemented disks. (Plate 5) Finally, the prepared test samples were stored in a humidor.

B. Fabrication of the Fifty, Seventy-five and One hundred-fifty Micron Test Samples

A method of producing an increasing series of marginal gaps was developed by placing circular stainless steel shims between the quartz disks prior to cementation. In order to fabricate standard sized stainless steel shims and to minimize surface burring, the following method was developed: (1) a core borer which was l0 mm in diameter was used to make a faint impressions upon the stainless steel sheets, (2) sharp iris scissors were used to cut the circular shims approximately 1.0 to 1.5 mm greater than the desired diameter, (3) the same core borer was used to make two 10 mm adhesive wax disks

Plate 4. The alignment fixture containing the quartz disks is positioned upon the precision press. A 14.7 newton load is applied to the center of the paired disks.

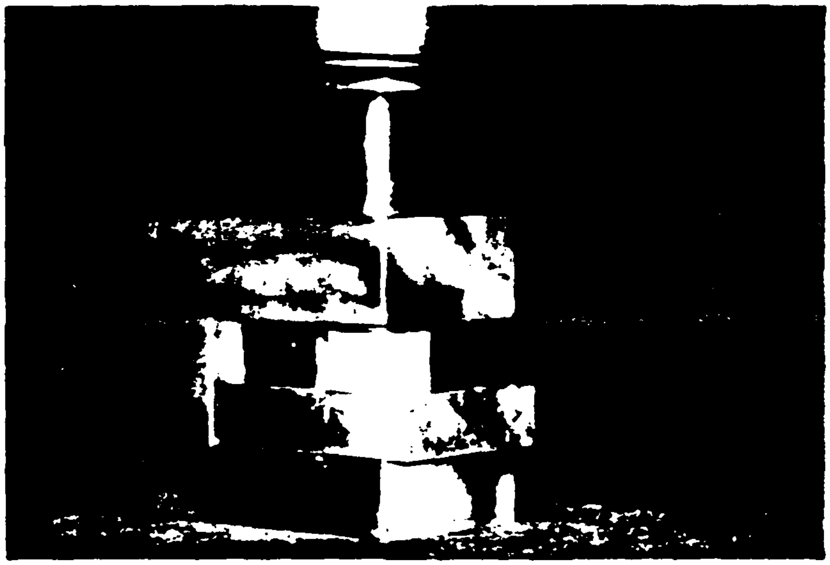
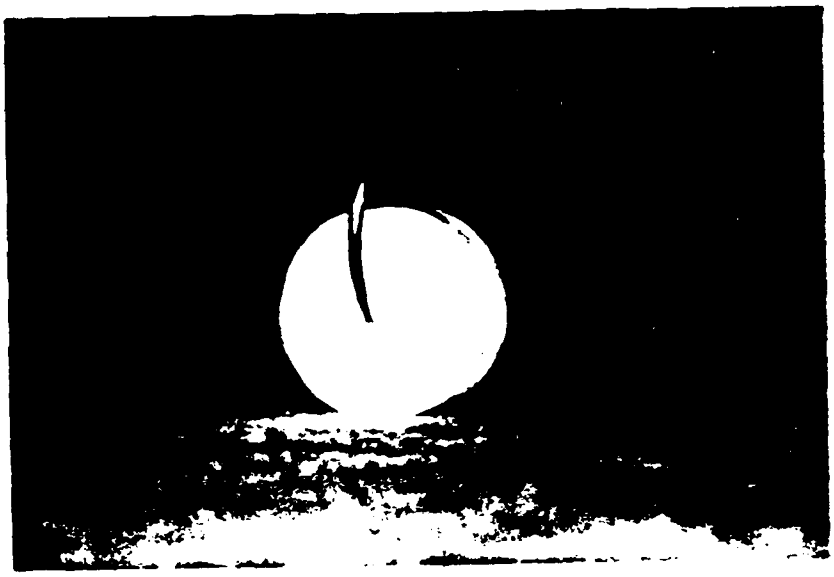


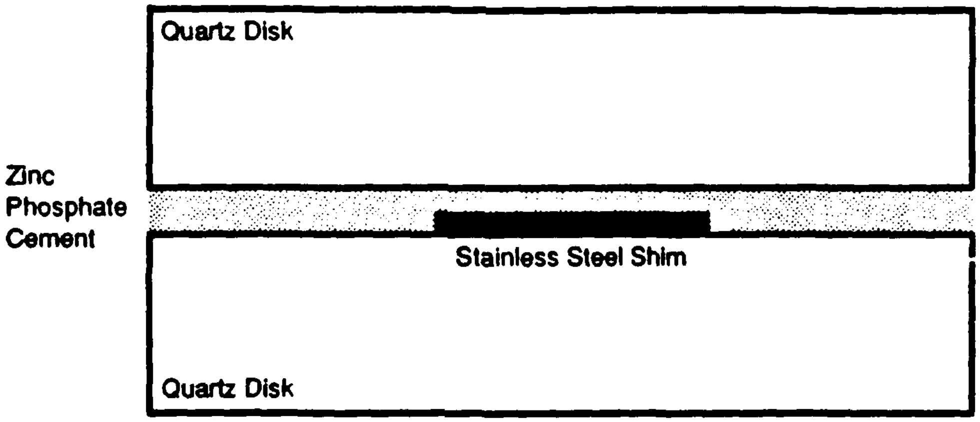
Plate 5. A stainless steel clamp was placed around the cemented test sample. The clamp for each test sample was engraved with the appropriate group letter and number.



which were centered on both sides of the oversized shims and sealed in place with sticky wax (Moyco Industries Inc., Philadelphia, Pa. 19123), (4) a piece of rope wax (Moyco Industries Inc., Philadelphia, Pa. 19123) was attached to the center of the shim with sticky wax, (5) the wax protected shim was immersed into a solution of aqua regia (1 part nitric acid and 3 parts hydrochloric acid) which eroded the exposed stainless steel, (6) the shim was then removed from the aqua regia and washed in distilled water, (7) the protective adhesive wax was removed and the shim was placed into xylene which aided in the removal of any wax residue, (8) the shim was washed in distilled water, dried, and its thickness was measured using a micrometer so as to insure that the original thickness has been maintained. As a result of this procedure, the shims which were created in the appropriate sizes and their edges were free of burrs.

The 50 micron test sample was created by inserting a 25 micron stainless steel shim between the quartz disks prior to its cementation. The shim was centrally positioned on one disk and the opposite disk, which contained the zinc phosphate cement, was carefully placed upon it. The test sample was positioned in the alignment fixture and placed in the precision press which was loaded in the standard manner. After 15 minutes the disk was removed from the precision press and post-cementation measurements were made. The combination of the 25 micron film thickness of the zinc phosphate cement and the 25 micron shim produced cement margins in the 50 micron range. The 75 and 150 microns test samples were created using the aforementioned procedure, however, 50 micron and 125 micron shims respectively were employed. *(Figure 1)*

Figure 1. Diagramatic representation of the lateral view of a test sample demonstrating the incorporation of a stainless steel shim.


Quartz Disk
Zinc
Phosphate
Cement
Stainless Steel Shim
Quartz Disk

C. Test Solution

Previous studies indicated that a dilute solution of lactic acid could be used as an appropriate in vitro test solution. (Beech 1983; Mesu I983) It was determined from pilot studies that a test solution of 0.1 molar lactic acid (15 ml of 85 percent lactic acid and 985 ml of distilled water) would be used as the dissolving medium.

D. Phase 1 - The Diffusion Study

The static dissolution phase of this study required the fabrication of a total of thirty-two test samples which consisted of eight 25, 50, 75, and I50 micron test samples. A group letter and number was engraved upon the supporting stainless steel clamp of each test sample. The 25 micron group was designated Group A and the samples within this group were numbered from 1 to 8. (Plate 6) The 50 micron group was designated as Group B and also numbered from 1 to 8. (Plate 7) The 75 and I50 micron test samples were designated as Group C and Group D respectively and numbered from 1 to 8.

A clear plastic container which was 590 mm long, 305 mm wide, and 100 mm deep was employed to contain the test solution of lactic acid. Five stainless steel rods which were 400 mm in length were placed into slots carved in the edges of the plastic container. Using a computer generated random sequence, seven test samples were arranged 25 mm apart on four of the support rods and the remaining four test samples were placed on the last support rod. The test samples were suspended from these stainless steel rods by hooks which were fashioned from orthodontic wire. (Plate 8)

The test samples were completely immersed in 6 liters of the lactic acid test solution and a plastic cover was placed over the container to reduce evaporation. The test solution and the test samples were left undisturbed until

Plate 6. Group A (25 micron) test sample.

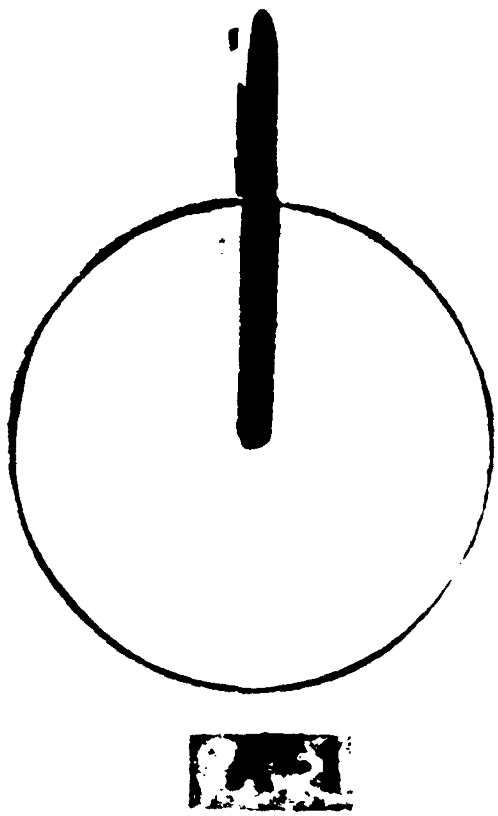


Plate 7. Group B (50 micron) test sample. The dark area which is centrally located is the 25 micron stainless steel shim.

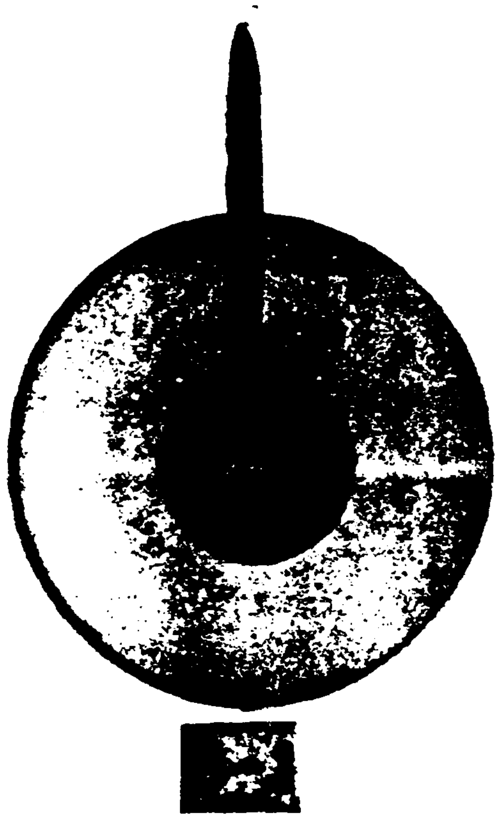
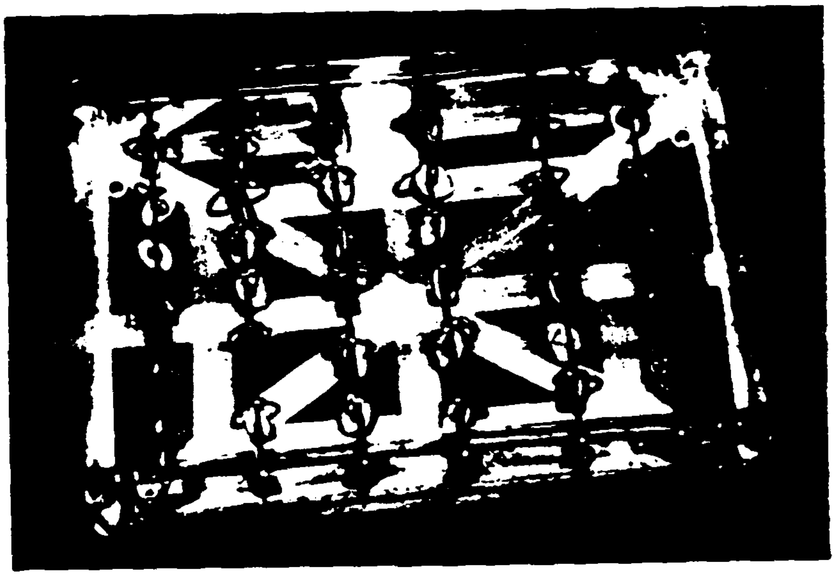


Plate 8. The thirty-two test samples suspended in the test solution for the Phase 1 Diffusion study.



cement dissolution on any one of the test samples came within two millimeters of the stainless steel shim. The investigator was able to visually monitor the degree of cement dissolution because of the container's transparent walls. Thereafter, the samples were removed from the test solution and they were photographed to enable a measurement of the cement dissolution.

The photography of the test samples was completed in a standardized manner. The camera body (Nikkon F-3), lens (Micro-Nikkor 105), and extension tube (PK-3 Nikkon) was mounted upon a copy stand and the lens was placed at maximum extension. In order to focus the test samples, the camera could be moved up and down upon the copy stand; however, once the correct focal distance was obtained, the camera remained stationary because all of the test samples were of the same diameter. Panatomic-X (Kodak) film was used and the standardized film plane magnification was 0.78X. A Bright Box (Bartholomew Mnufacturing Incorporated, Des Planes, Illinois, 60017) provided the background illumination. A twelve inch square piece of plexiglass, with a two inch slit which permitted the clamped samples to lie flat, was used to support the test sample. The photograph of each samples was identified by placing the appropriate group letter and number under the test sample. All of the test samples were photographed under the aforementioned conditions by the same photographer, the prints were processed in a standard manner, and 8" x 10" enlargements were made. (Plate 9) The area of the remaining cement was measued from the photograph using a Zeiss Interactive Digital Analysis System. (Carl Zeiss, Inc., Thornwood, N.Y.) (Plate 10) A one way analysis of variance was selected as the statistical test of choice for comparing the areas of remaining cement among the groups.

Plate 9. A post-dissolution test sample (B-1). This test sample demonstrates the centrally located unaffected cement core and the outer "halo" of affected cement.

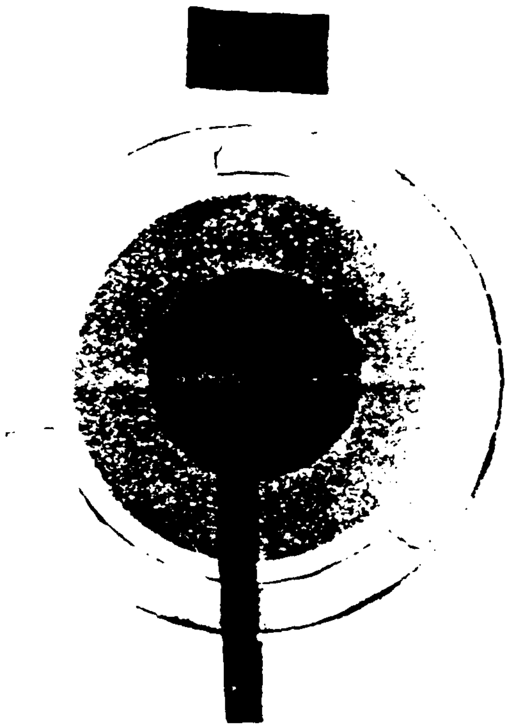
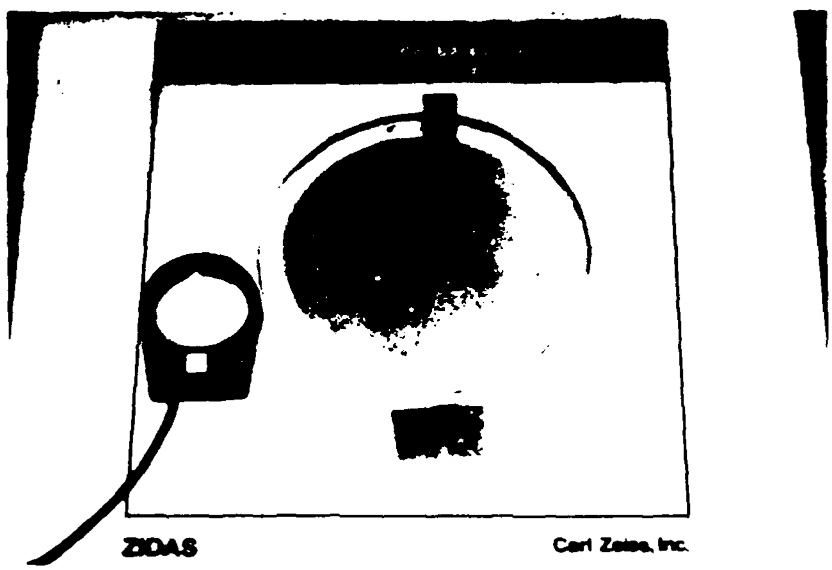


Plate 10. A Zeiss Interactive Digital Analysis System measuring the remaining cement area in a standard photograph of a post-dissolution test sample.


ZIDAS
Carl Zeiss, Inc.

After photographs were made of the test samples, the stainless steel clamps were removed from the test samples and the quartz disks were carefully separated. Utilizing a dissecting microscope, the unaffected cement core and the affected "halo" layers were separated from one another. (Plate 9) The cement collected from the two layers were individually examined using x-ray diffraction techniques.

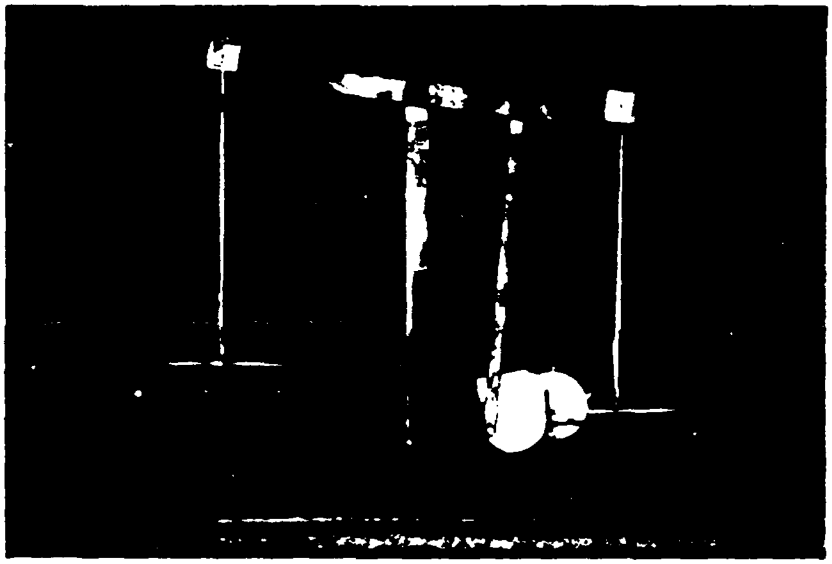
E. Phase 2- The Dynamic Study

While Phase 1 of this study considered the effects of simple diffusion upon luting cement solubility in a static solution, Phase 2 of this study addressed the influence of fluid flow on solubility since the oral cavity is a dynamic environment and luting cements are subjected to multiple sources of fluid flow.

A specially designed mechanical apparatus, the "Windeler" machine, was built to impart cyclic motion to the test samples. (Plate 11) The test samples were attached to two horizontal cross-bars which moved in a vertical direction. The vertical movement of the test samples was equivalent to 70 mm and this movement was termed "one cycle." Fourteen cycles were completed in one minute.

Four groups of test samples were fabricated, measured, and identified in the same manner as in Phase 1. Group A represented the 25 micron control group; while Group B, Group C, and Group D represented the 50, 75, l50 micron samples respectively. To facilitate the random placement of the test samples upon the horizontal cross-bars, each group consisted of five test samples. The twenty samples were attached to the cross-bars with hooks fabricated from orthodontic wire. Additionally, the test samples were secured to

Plate 11. Mechanical apparatus employed in the Phase 2 Dynamic study. This machine move the test samples in a cyclic, vertical direction.

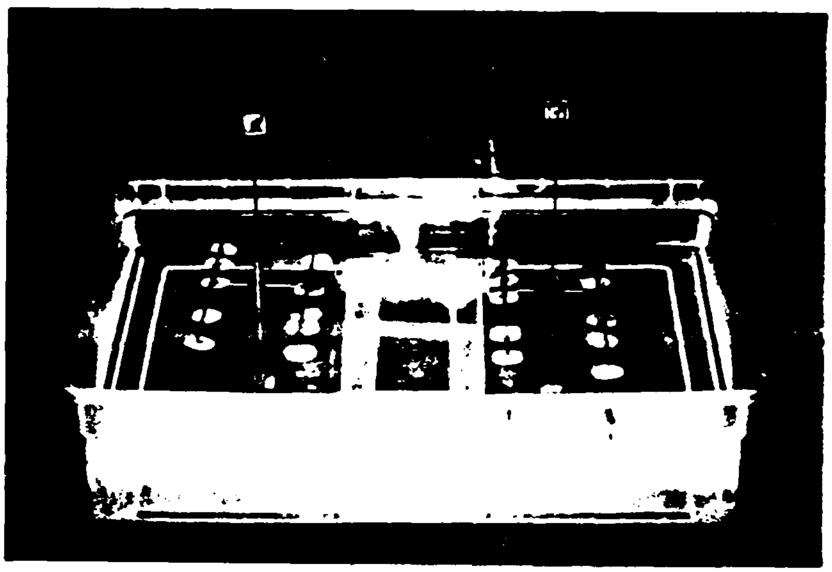


the orthodontic hooks with sticky wax so as to prevent dislodgement of the test samples during the dynamic phase of the study.

The suspended samples were centered over the plastic container which contained ten liters of the lactic acid test solution. This arrangement permitted the test samples to remain completely submerged throughout the investigation. (Plate 12) The dynamic phase of this study commensed with the start of the "Windeler" machine, and continued until cement dissolution was within two millimeters of the shim of any one of the test samples.

At the end of the test period the samples were removed from the test solution and photographed in the same manner as in Phase 1. The area of remaing cement for each test sample was measure using the Zeiss Interactive Digital Analysis System. A one way analysis of variance was selected as the statistical test of choice for comparing the remaining cement area among the groups.

Plate 12. Test samples suspended from the horizontal crossbars of the mechanical apparatus employed during the Phase 2 Dynamic study.



IV. RESULTS

This study was divided into two phases: Phase 1 investigated luting cement solubility as it relates to diffusion and marginal gap size while Phase 2 evaluated the influence of a dynamic environment on luting cement solubility.

A. Phase 1 - The Diffusion Study

The diffusion study was terminated 30 days after the test samples were immersed in the test solution since this was when cement dissolution reached within 2 mm of any one of the test samples' shims. The mean and standard deviation of the marginal openings of the test samples are listed in Table 1. The Phase 1 means and standard deviations for the cement remaining after dissolution are listed in Table 2. From the (ANOVA) it was determined that there was a statistically significant difference among the Phase 1 groups. ($p<0.001$) (Table 3) A Duncan's multiple comparison test was employed to test for differences between pairs of means. This test demonstrated that the cement areas remaining after dissolution in the Group D test samples were statistically smaller than the cement remaining in Groups A, B, and C. ($p<0.05$) The Duncan's multiple comparison test also demonstrated that there was no statistically significant difference between the remaining areas of cement in the Group A, B, and C test samples. ($p<0.05$) The data for the Phase 1 diffusion study is located in Appendix B.

B. Phase 2 - The Dynamic Study

During the Phase 2 study the cement reached the 2 mm point within 7 days, this indicated a dissolution rate higher than that observed in the Phase 1

TABLE 1. PHASE 1 MEAN CEMENT MARGINS

Group	N	Mean (Microns)	Standard Deviation
A	8	24.50	2.07
B	8	51.75	2.08
C	8	74.00	3.29
D	8	148.00	4.18

TABLE 2. MEAN REMAINING CEMENT AREAS OF THE PHASE 1 TEST SAMPLES

Group	N	Cement Area (Square mm)	Standand Deviation
A	8	12,382	609.51
B	8	12,664	460.90
C	8	12,244	316.85
D	8	9876	475.37

TABLE 3. SUMMARY TABLE FOR A ONE-WAY ANALYSIS OF VARIANCE COMPARING THE AVERAGE CEMENT AREA REMAINING FOR EACH PHASE 1 TEST SAMPLE

Source	DF	SS	MS	F- value	F- probability
Group	3	39900769.09	13300256.36	58.44	0.001
Error	28	6372323.12	227582.96		

part of the study. The mean and standard deviation of the marginal openings of the test samples are listed in Table 4. The Phase 2 means and standard deviations for the cement remaining after dissolution are listed in Table 5. A (ANOVA) demonstrated a statistically significant difference among the four groups. ($p<0.001$) (Table 6) A Duncan's multiple comparison test demonstrated that the cement remaining in the test samples of Group D was statistically smaller than the cement remaining in Groups A, B, and C. ($p< 0.05$) A statistically significant difference between Groups A, B, and C was not noted. ($p<0.05$) The data for the Phase 2 dynamic study is located in the Appendix B.

TABLE 4. PHASE 2 MEAN CEMENT MARGINS

Group	N	Mean (Microns)	Standard Deviation
A	5	24.8	3.11
B	5	53.2	2.48
C	5	73.2	3.27
D	5	149.6	6.65

TABLE 5. MEAN REMAINING CEMENT AREA OF THE PHASE 2 TEST SAMPLES

Group	N	Cement Area (Square mm)	Standard Deviation
A	5	17,314	167.27
B	5	17,232	503.25
C	5	16,956	496.80
D	5	14,428	978.37

TABLE 6. SUMMARY TABLE FOR A ONE-WAY ANALYSIS OF VARIANCE COMPARING THE AVERAGE CEMENT AREAS REMAINING FOR EACH PHASE 2 TEST SAMPLE

Source	DF	SS	MS	F- value	F-Probability
Group	3	28500110.15	9500036.71	25.5844	0.001
Error	16	5941136.80	371321.05		

V. DISCUSSION

The rate at which dental luting cements dissolve in the oral environment is crucial to the long-term success of the cemented cast restoration. Thus, solubility is probably the most significant clinical physical property of luting cements. (Phillips 1973) Various authors have stated that the rate of cement solubility is directly related to the degree of luting cement exposed to the oral environment. (Cooper 1970; Johnston 1971) These results, however, conflict with Fick's First Law of Diffusion. This physical law states that the rate of dissolution due to diffusion is independent of the mass of exposure. (Adams 1956) Furthermore, the rate of diffusion of a neutral solute across a unit area is determined by the diffusion constant of the solute and the concentration gradient. (Williams 1973) (Figure 2) Therefore, Fick's law would predict that the rate of luting cement dissolution is independent of its exposed mass. It should be noted, however, that Fick's law applies only to dissolution which results from diffusion and cement exposed to abrasive forces or convective motions would not be expected to be subject to this law.

In general, the placement of supragingival margins has been recommended. (Shillingburg 1987) However, the placement of subgingival margins may be necessary due to caries, esthetic requirements, defective restorations, and the need for additional resistance and retention. Since subgingival margins and their corresponding cement lines are in a relatively stagnant area (Beech and Bandyopahyay 1983), the solubility of the exposed luting cement may be significantly influenced by simple diffusion (Smith 1982)

Figure 2. Diagramatic representation of the dissolution of dental luting cement as it relates to Fick's First Law of Diffusion. Fick's First Law of Diffusion would predict that the rate of cement dissolution would be the same for both cement margins regardless of the fact that cement line B has twice the exposed area. This is because B has twice the available cement to resist dissolution. Hence, the rate of dissolution due to diffusion would be the same for cement lines A and B.

Fick's First Law of Diffusion $= \frac{dm}{dt} = -D\ (A)\ \frac{dc}{dx}$

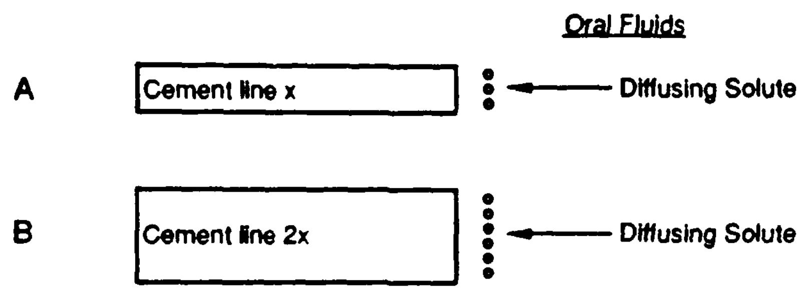
$\frac{dm}{dt}$ = rate at which m grams of solute cross the reference plane

$\frac{dc}{dx}$ = concentration gradient*

D = Diffusion constant of the solute*

A = cross-sectional area in which solute is diffusing

* Determinates of the rate of diffusion


Oral Fluids
A
Cement line x
Diffusing Solute
B
Cement line 2x
Diffusing Solute

and Fick's First Law of Diffusion may be applicable for predicting the rate of cement dissolution in these areas.

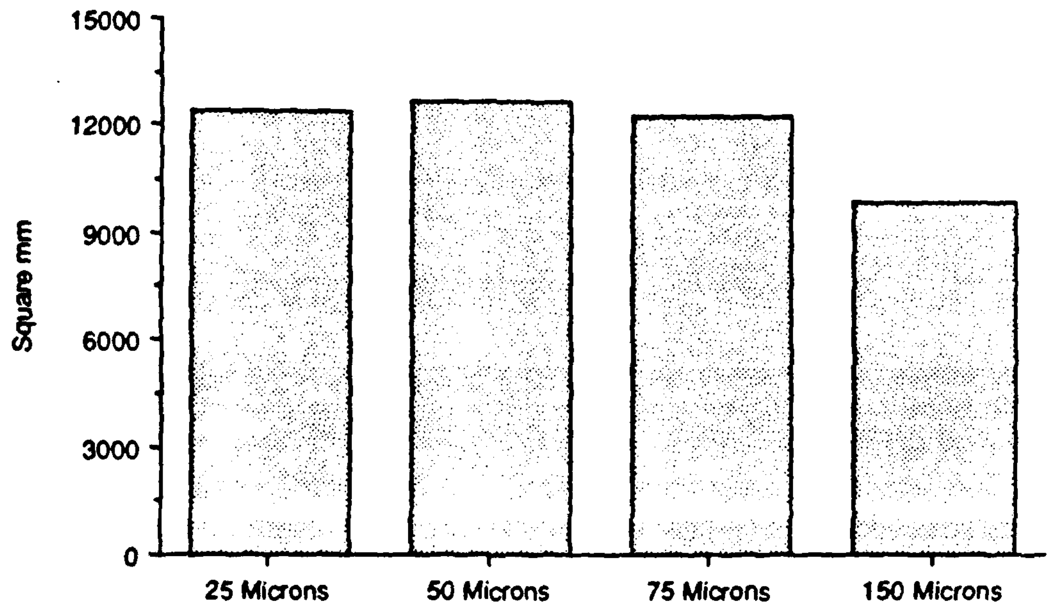
Presently, the exact relationship between the marginal gap and the rate of cement dissolution is not known. (Smith 1982) To more closely evaluate luting cement solubility in the manner and dimensions in which they are used, a new in vitro experimental model was developed. This model permits the direct visualization of the luting cement as it dissolves, and addresses the question of how the rate of cement dissolution is influenced by the degree of marginal opening.

Type 1 zinc phosphate cement was selected as the luting agent in this investigation because it is the standard against which other cements are compared and because of its long history of clinical use. Moreover, recent studies have demonstrated that it is still the most widely used of the luting cements. (Smith 1976; Abelson 1980)

The results from Phase 1, the diffusion study, demonstrated that there was no statistically significant difference in the rate of cement solubility between the 25, 50, and 75 micron test samples. This was determined by examining and measuring photographs which were made of each of the test samples. These results support Fick's First Law of Diffusion because, as the law predicts, cement dissolution was independent of the size of the exposed cement line. Moreover, the driving forces involved in cement dissolution due to diffusion were the diffusion constant of the cement solute and the concentration gradient.

The 150 micron test samples, however, demonstrated a slight increase in cement dissolution which was statistically significant. (Figure 3) An examination of the photographs of the 150 micron test samples revealed that

Figure 3. Phase 1 Diffusion Study - Bar chart of the mean areas of cement remaining after dissolution.


15000
12000
9000
6000
3000
0
Square mm
25 Microns
50 Microns
75 Microns
150 Microns

areas of the luting cement were separating away from the cement core. Moreover, this occurred along the bottom of the test samples. These results suggest that the dissolution of the cement was not only dependent upon diffusion but that erosion may also be occurring. Since the gap between the quartz disks is relatively large, the eroded particles of cement fell from the cement core. It should be noted that the test samples were suspended vertically in the test solution. Due to this physical orientation, gravitational forces may have assisted in the erosion along the bottom of the test samples. Thus, the decreased structural integrity of the luting cement caused by diffusion and the influence of gravitational forces may have contributed to the separation of the eroded cement from the cement core.

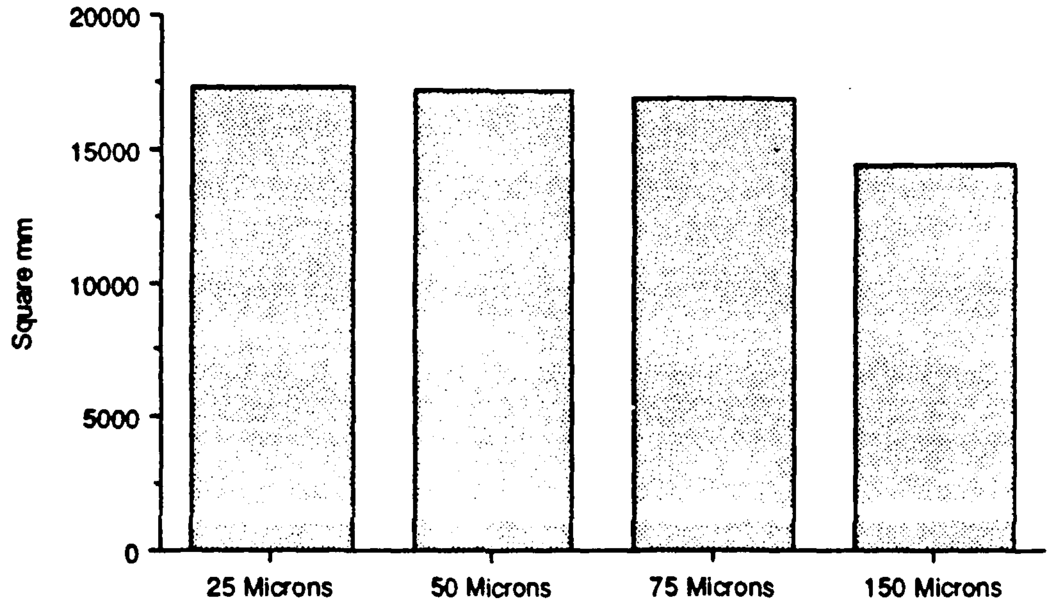
During Phase 1, test samples were exposed to a static test solution so as to evaluate the influence of simple diffusion upon luting cement solubility. The oral cavity, however, is a dynamic environment and dental luting cements are subject to multiple sources of fluid flow. (Beech 1983) Phase 2 of this study investigated the influence of a dynamic environment upon the solubility of zinc phosphate cement. As in Phase 1, the 25, 50, and 75 micron groups did not demonstrate any statistically significant differences. However, there was evidence that the mean dissolution of the cement slightly increased as the marginal gaps increased in size from 25 to 75 microns. Moreover, the 150 micron test samples had significantly more cement dissolution than the other three groups. The results suggest that the increase in the rate of cement dissolution was due to diffusion and forced convection of the solute because the vertical movement of the test samples distributed the solute from the exposed cement and thereby caused the cement to release more solute. Thus, forced convection played an important role in increasing the rate of dissolution. As a

result, this investigation required one-fourth the time and demonstrated a tenfold increase in the rate of cement dissolution when compared to the Phase 1 results. When the rate of cement dissolution for the 25, 50, and 75 micron test samples were compared, it was found that there were no statistically significant differences between these groups. This suggests that when the marginal gap is small diffusion may be the major mechanism of cement dissolution, and losses due to convection may be independent of the small gap. The 150 micron group, however, demonstrated an increased amount of cement dissolution when compared to the smaller marginal gaps. (Figure 4) This suggests that the150 micron gap size was large enough to allow small amounts of convection to occur within the gap. The effects of convecticn within the larger gap combined with diffusion may have significantly increased the dissolution rate.

Type 1 zinc phosphate cement has a 25 micron film thickness. While restorations with marginal gaps of such small dimensions should be a goal of the practitioner, they are seldom achieved in clinical practice. (Roberts 1973) Christensen has demonstrated that subgingival marginal gaps as large as 120 microns may be judged acceptible. Supragingival margins as small as 40 microns, however, were judged clinically unacceptible. Thus, when access and visibility are limited, the subgingival marginal discrepancies and subsequent cement lines may be relatively large when compared to the supragingival margin.

The subgingival margin is difficult to finish since it is inaccessible. Similarly, this lack of access decreases the abrasive effects from tooth brushing and the mastication of food in this marginal area. Because of the limited

Figure 4. Phase 2 Dynamic Study - Bar chart of the mean areas of cement remaining after dissolution.


20000
15000
10000
5000
0
Square mm
25 Microns
50 Microns
75 Microns
150 Microns

influence of abrasion, subgingivally luting cement dissolution may be caused primarily by diffussion.

The supragingival margin, however, is more easily evaluated and finished. The marginal discrepancy of these accessible margins can be decreased as much as 30-40 microns when proper finishing techniques are employed. (Krug and Markley 1969; Kishimoto et. al. 1981) Hence, the margins which are most susceptable to the effects of abrasion from tooth brushing and the mastication of food can be minimized by finishing procedures. This reduction in the area of the exposed luting cement line will decrease the effects of abrasion on the cement and enhance the longevity of the restorations.

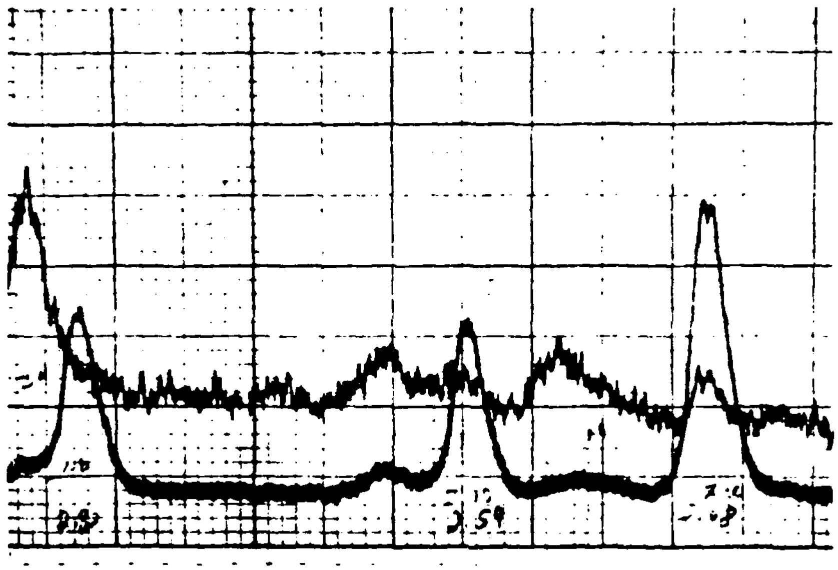
As demonstrated by the results of this investigation, the dissolution of the dental luting cement between the 25, 50, and 75 micron test samples did not significantly differ during the Diffusion Study and a similar finding was noted for the Dynamic Study. It was only when the degree of marginal opening was increased to 150 microns that significant differences occurred. While it has been suggested that the film thickness of luting cements should be used as a guide for evaluating marginal discrepancies, the results of this study suggest that the minimal marginal gap for Type 1 zinc phosphate cement may be increased threefold without significant differences in terms of cement dissolution. While this investigator does not advocate the use of less than optimal margins, these results do provide one explanation as to why there is still the possibility for clinical success even though the restoration lacks optimal marginal adaptations. Moreover, this study may provide an explanation as to why catastrophic failures due to cement dissolution are not more frequently observed.

During the Phase 1 diffusion study, each of the test samples evidenced two distinct layers of cement. The outer layer of cement which was closest to the test solution averaged 0.5 mm to 1.0 mm in width. This layer of the luting cement, which was termed the "halo," encircled the remaining cement core. (Plate 9) The "halo" consisted of cement which was partially dissolved by the test solution. In order to determine whether there was a difference in the chemical compostion of the affected "halo" layer and the unaffected cement core, x-ray diffraction studies were performed on both of these layers. The chemical compostion of the unaffected cement core and a standard mix of zinc phosphate cement was also compared via x-ray diffraction. (Appendix A)

The matrix of zinc phosphate cement has been described as an amorphous zinc alumino-phosphate gel with unreacted powder particles which are 2 to 8 microns in diameter. (Servais and Cartz 1971; Smith 1982) The x-ray diffraction study, however, revealed that zinc oxide was absent from the affected "halo" layer. (Plate 13) These results suggest that the unreacted zinc oxide particles are among the first components of the set zinc phosphate cement to be displaced by dissolution; thereby confirming the prediction of previous investigators. (Wilson 1970; Servais and Cartz 1971; Beech and Bandyopahyay 1983)

X-ray diffraction also revealed that the unaffected core of the zinc phosphate test samples contained a crystalline reaction component. An analysis of this inner zone of cement suggested that the crystalline component increased with time since the largest peaks were observed in specimens that were submerged in lactic acid for 30 days. As a general rule, with the exception of zinc oxide powder, a standard mix of zinc phosphate cement does not contain a crystalline component. One possible explanation for the

Plate 13. The X-ray diffraction record. The bottom tracing is from the unaffected cement core and demonstrates the three peaks which represent zinc oxide. The upper tracing, from the affected or "halo" layer, demonstrates that zinc oxide is no longer present.



crystallization of the luting cement in the test samples was that when the quartz disks were cemented together, excess water which would normally have evaporated from an exposed test sample was trapped and therby initiated the crystalline formation. Similar crystal formation might be expected during the cementation of a cast crown onto a prepared tooth. (Servais and Cartz 1971) Therefore, the discovery of cement crystallization is significant because this crystallization process destroys adhesive bonds which may form between the tooth and the casting. (Wilson 1978) Consequently, when utilizing zinc phosphate cement, the clinician must rely upon the geometric form of the preparation design and the mechanical irregularities present on the tooth and castings for retention of the cast restoration.

VI. SUMMARY

This investigation was designed to examine the rate of dental luting cement dissolution as it relates to the degree of marginal gap opening. The study consisted of two parts: Phase 1 evaluated the effect of diffusion upon cement dissolution while Phase 2 investigated the effects of a dynamic environment. In both phases, test samples were fabricated to expose 25, 50, 75, and 150 microns of zinc phosphate cement lines to a test solution of 0.1 molar lactic acid. The rate and amount of cement dissolution was visible since the test samples were comprised of luting cement interspersed between transparent quartz disks. Standard photographic records were made at the end of both test periods for all of the test samples. The photographs were then measured with the Zeiss Interactive Digital Analysis System to statistically evaluate the area of remaining cement. Additionally, since the remaining cement in each of the test samples following the diffusion study demonstrated a "halo" of affected cement which surrounded the cement core, an x-ray diffraction study to evaluate the chemical composition was conducted upon both layers.

The following results and conclusions may be drawn from this investigation.

(1) The Phase 1 diffusion study demonstrated that the rate of luting cement solubility was not statistically different when the marginal opening were 25, 50, and 75 microns in size. The 150 micron marginal gap, however, did demonstrate a slight increase in cement dissolution which was statistically significant.

The lack of statistically significant differences in cement solubility between the 25 to 75 micron test samples corresponds with the results anticipated by Fick's First Law of Diffusion. Fick's law maintains that dissolution due to diffusion is independent of the size of exposure. The variance found in the 150 micron test samples may have resulted from a combination of factors, including diffusion and erosion. The erosion probably was due to the large size of the marginal gap and the impact of gravitational forces.

(2) The Phase 2 dynamic study revealed no statistically significant difference in the rate of cement dissolution at the 25, 50, and 75 micron levels. The 150 micron group, once again, demonstrated a slight increase in cement dissolution which was statistically significant.

The results from the dynamic study suggests that the vertical movement of the test samples through the test solution created convection forces which contributed to the increased rate of cement dissolution. The 25, 50, and 75 microns test samples demonstrated no statistically significant difference in their rates of dissolution. This suggests that when the gap size is small diffusion may be the primary mechanism of dissolution. The 150 microns test samples, however, demonstrated an increased rate of dissolution. This suggests that the gap size for this test sample may be large enough to allow convection forces to occur within the gap. The convective flow of solute and solvent within the gap may represent a mechanical effect which enhances the erosion of the cement .

(3) During both the Phase 1 and Phase 2 investigations, the 25, 50, and 75 micron test samples demonstrated little or no statistically significant differences in the dissolution of the luting cement. While this investigator concurs with the proposition that a cement's film thickness should serve as a guide for evaluating the maximum amount of marginal discrepancy, the results of this study suggests

that the minimal marginal gap for Type 1 zinc phosphate cement may be increased threefold without significantly affecting cement solubility from the restoration. Once the marginal opening reaches 150 microns in width, however, there is a marked increase in the dissolution of the luting cement. This investigator merely suggests that the results of this investigation may provide one explanation as to why restorations with less than optimal marginal adaptations may still demonstrate clinical success.

(4) The x-ray diffraction study of the "halo" layer of the luting cement revealed a lack of zinc oxide. This finding suggests that the unreacted zinc oxide particles may be among the first components displaced from the cement. X-ray diffraction of the unaffected cement core, however, demonstrated a retention of the zinc oxide particles and the other chemical constituents which are present in a standard mix of zinc phosphate cement.

Unlike a standard mix of zinc phosphate cement, the cement core of these test samples revealed a crystalline component which probably resulted from excess water being trapped between the quartz disks. This finding is significant because it provides an additional explanation for zinc phosphate cement's lack of adhesion.

APPENDIX A

X-RAY DIFFRACTION TECHNIQUE EMPLOYED IN THIS INVESTIGATION

X-RAY DIFFRACTION TECHNIQUES

X-ray diffraction is a technique which can be used to identify crystalline structures. Generally a monochromatic incident x-ray beam is directed at the test sample and a portion of the diffracted beam will be scattered without energy loss or gain. The distribution of the scattered intensity is related to the crystalline nature of the test sample. The position, width and intensity of the x-ray diffraction lines which are produced by the specimen are scanned by a detector and are directly recorded. It is from this information that the internal crystalline structure of the test sample can be ascertained.

In this study a Siemens Kristalloflex 4 x-ray generator and a Siemens Goniometer (Diffractometer) Type F was utilized. The anode material for this x-ray diffraction tube was copper, the wavelength was 1.541 angstroms, and a nickel filter slide was utilized. The recorder paper speed was set at one-half inch per minute. The rotational speed for the detector was one-half degree per minute and the amount of its rotation was 0 degrees to 90 degrees.

APPENDIX B

DATA FOR TEST SPECIMENS

TABLE 7. THE MARGINAL GAP DATA FOR TEST SAMPLES A (25 microns)

THE PHASE 1 DIFFUSION STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
A-1	12.812	12.838	0.026
A-2	12.563	12.588	0.025
A-3	12.672	12.695	0.023
A-4	12.515	12.537	0.022
A-5	12.542	12.565	0.023
A-6	12.656	12.684	0.028
A-7	12.733	12.756	0.023
A-8	12.485	12.511	0.026

The mean cement margin created for the Series A test samples was 0.0245 mm or 24.5 microns.

TABLE 8. THE MARGINAL GAP DATA FOR TEST SAMPLES B (50 microns)
THE PHASE 1 DIFFUSION STUDY

	Pre-cementation Mean (millimeters)	Pcst-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
B-1	12.487	12.536	0.049
B-2	12.425	12.477	0.052
B-3	12.249	12.304	0.055
B-4	12.492	12.542	0.050
B-5	12.737	12.784	0.047
B-6	12.530	12.580	0.050
B-7	12.569	12.623	0.054
B-8	12.700	12.755	0.055

The mean cement margin created for the Series B test samples was 0.05175 mm or 51.75 microns.

TABLE 9. THE MARGINAL GAP DATA FOR TEST SAMPLES C (75 microns)

THE PHASE 1 DIFFUSION STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
C-1	12.769	12.846	0.077
C-2	12.723	12.793	0.070
C-3	12.491	12.567	0.076
C-4	12.786	12.857	0.071
C-5	12.690	12.762	0.072
C-6	12.839	12.910	0.071
C-7	12.823	12.900	0.077
C-8	12.882	12.960	0.078

The mean cement margin created for the Series C test samples was 0.074 mm or 74 microns.

TABLE 10.THE MARGINAL GAP DATA FOR TEST SAMPLES D (150 microns) THE PHASE 1 DIFFUSION STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
D-1	12.452	12.595	0.143
D-2	12.781	12.935	0.154
D-3	12.717	12.863	0.146
D-4	12.653	12.806	0.153
D-5	12.151	12.303	0.152
D-6	12.498	12.643	0.145
D-7	12.864	13.012	0.148
D-8	12.236	12.379	0.143

The mean cement margin created for the Series D test samples was 0.148 mm or 148 microns.

TABLE 11. PHASE 1 DIFFUSION STUDY: MEAN REMAINING CEMENT AREAS FOR EACH OF THE TEST SAMPLES AS MEASURED IN SQUARE MILLIMETERS FROM STANDARD PHOTOGRAPHS.

SPECIMEN	GROUP A	GROUP B	GROUP C	GROUP D
1	12,576	13,080	11,690	9,911
2	11,943	12,636	12,600	9,086
3	13,111	13,000	12,456	10,090
4	12,453	12,330	12,110	9,720
5	11,543	12,665	12,433	9,744
6	11,626	12,756	12,120	9,541
7	12,980	11,730	11,996	10,660
8	12,830	13,120	12,550	10,253
MEAN:	12,382	12,664	12,244	9,876

TABLE 12. THE MARGINAL GAP DATA FOR TEST SAMPLES A (25 microns)
THE PHASE 2 DYNAMIC STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
A-1	12.739	12.762	0.023
A-2	12.671	12.692	0.021
A-3	12.558	12.586	0.028
A-4	12.538	12.566	0.028
A-5	12.821	12.835	0.024

The mean cement margin created for the Series A test samples was 0.0248 mm or 24.8 microns.

TABLE 13. THE MARGINAL GAP DATA FOR TEST SAMPLES B (50 microns)
THE PHASE 2 DYNAMIC STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
B-1	12.529	12.582	0.053
B-2	12.570	12.620	0.050
B-3	12.255	12.312	0.057
B-4	12.483	12.536	0.053
B-5	12.425	12.478	0.053

The mean cement margin created for the Series B test samples was 0.0532 mm or 53.2 microns.

TABLE 14. THE MARGINAL GAP DATA FOR TEST SAMPLES C (75 microns) THE PHASE 2 DYNAMIC STUDY

	Pre-cementation Mean (miilimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
C-1	12.687	12.760	0.073
C-2	12.666	12.740	0.074
C-3	12.493	13.065	0.072
C-4	12.887	12.956	0.069
C-5	12.846	12.924	0.078

The mean cement margin created for the Series C test samples was 0.0732 mm or 73.2 microns.

TABLE 15.THE MARGINAL GAP DATA FOR TEST SAMPLES D (150 microns)
THE PHASE 2 DYNAMIC STUDY

	Pre-cementation Mean (millimeters)	Post-cementation Mean (millimeters)	Mean Cement Margin (millimeters)
D-1	12.500	12.648	0.148
D-2	12.157	12.314	0.157
D-3	12.722	12.867	0.145
D-4	12.458	12.600	0.142
D-5	12.864	13.020	0.156

The mean cement margin created for the Series D test samples was 0.1496 mm or 149.6 microns.

TABLE 16. PHASE 2 DYNAMIC STUDY: MEAN REMAINING CEMENT AREAS FOR EACH OF THE TEST SAMPLES AS MEASURED IN SQUARE MILLIMETERS FROM STANDARD PHOTOGRAPHS.

SPECIMEN	GROUP A	GROUP B	GROUP C	GROUP D
1	17, 180	17,640	17,500	14,690
2	17,500	17,680	16,470	14,850
3	17,380	17,180	16,660	14,900
4	17,100	17,230	16,669	15,010
5	17,410	16,430	17,490	12,690
MEAN:	17,314	17,232	16,956	14,428

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VITA

Michael Steven Jacobs was born in Houston, Texas, on July 18, 1950; the son of Charles Edward Jacobs and Mary Jane Jacobs. He graduated from the University of Houston in June l973 with a Bachelor of Science in Biology; and, entered the University of Texas Dental School at Houston in July of 1973. At that time he received an Air Force Health Professional Scholarship and was commissioned as a Second Lieutenant. He received his Doctor of Dental Surgery degree in May of 1977.

In June of 1977, he entered a General Practice Residency Program at Carswell Air Force Base and graduated in June of 1978. From July of 1978 to June of 1982 he served as a general dental officier at Vance Air Force Base, Enid Oklahoma. In 1982 he was assigned to Kelly Air Force Base,San Antonio, Texas, and assumed the position of dental laboratory officer. During August of 1984 he was selected as the first Air Force dentist to be assigned to the United States Tri-Service Commitment to Honduras.

In July of 1985 he entered the Post-Doctoral Prosthodontics Program at the University of Texas Health Science Center at San Antonio; and, the following year was admitted as a candidate for the Master of Science degree at the Graduate School of Biomedical Sciences. He was awarded first place in The American College of Prosthodontists First Annual Table Clinic Competition held in San Diego in October of 1987. He has been assigned as a Staff Prosthodontist at Sheppard, Air Force Base, Wichita Falls,Texas.

This thesis is submitted for partial fulfilment of a Master of Science degree to be awarded upon completion of requirements, May 1988, from the University of Texas Health Science Center at San Antonio School of Biomedical Sciences.

AN INVESTIGATION OF DENTAL LUTING CEMENT SOLUBILITY
AS A FUNCTION OF THE MARGINAL GAP

Michael Steven Jacobs, D.D.S.

The Universtiy of Texas Graduate School of Biomedical Sciences
at San Antonio

Supervising Professor: A. S. Windeler D.D.S., M.Sc., Ph.D.

Dental cements used in fixed prosthodontics have the primary purpose of luting or sealing the cast restoration to the prepared tooth. A permanent luting cement should have high strength, low film thickness, be non-irritating to the dental pulp, and have low solubility. A critical property of luting cement is its solubility in oral fluids. If the cement dissolves at an unacceptably high rate the tooth is susceptible to recurrent caries and the retention of the cast restoration can be compromised.

It has been stated that the rate of luting cement solubility is related directly to the size of the marginal gap that exists between the cast restoration and the prepared tooth. (Cooper 1971; Johnston 1971) Thus, the larger the

marginal gap, the larger the resultant cement line and the more rapid the rate of cement dissolution. Fick's First Law of Diffusion, however, would predict that cement solubility as it relates to diffusion is dependent on the diffusion constant of the solute and concentration gradients . This would indicate that cement solubility as it relates to diffusion is independent of the mass of cement exposed. Presently, the exact relationship between the marginal gap size and the rate of cement dissolution is not known.

The purpose of this study was to investigate the rate of cement solubility as it relates to the degree of marginal opening. Both a static and dynamic environment was used to evaluate the influence of diffusion and convection on cement solubility.

Standardized test samples were made that would simulate marginal gaps and subsequent cement lines. The dimensions of these marginal gaps were 25, 50, 75, and 150 microns. Type 1 zinc phoshate cement was used as the luting agent. Phase 1 of the study evaluated test samples that were placed in a static solution. This allowed the investigator to study cement dissolution as it related to diffusion. Phase 2 of the study investigated the influence of a dynamic enviroment on the dissolution of cement.

At the end of the test periods, standardized photographs were made of the test samples. These photographs were used to measure and record the remaining areas of cement so as to compare the rate of cement dissolution as it relates to the degree of marginal opening.

A one-way ANOVA for the Phase 1 Diffusion Study revealed that there were statistically significant differences between the test groups. A Duncan's Multiple Comparison Test demonstrated that the 150 micron test group had a small but statistically significant decrease in the remaining dental luting

cement. The 25, 50, and 75 micron groups demonstrated no statistically significant differences in the amount of remaining luting cement. ($p < 0.05$)

A one-way ANOVA was also performed on Phase 2 Dynamic Study. This test revealed that there were statistically significant differences among the test groups. A Duncan's Multiple Comparison Test demonstated that there was a statistically significant decrease in the remaining cement in the 150 micron test group. The remaining cement in the 25, 50, and 75 micron test groups were not significantly different. ($p < 0.05$)

During the Phase 1 Diffusion Study two distinct regions of luting cement were visible in the test samples. A small outer "halo" of cement which was termed the "affected layer" surrounded a larger inner "unaffected core" of luting cement. These layers of cement were evaluated using X-ray diffraction techniques. The results of these studies indicated that zinc oxide was absent in the outer affected layer of luting cement while present in the inner unaffected core of cement. This suggests that zinc oxide is one of the first constituents of zinc phosphate cement lost due to dissolution.

The results of the Phase1 Diffusion Study tend to support Fick's First Law of Diffusion when the marginal gap is less than 75 microns. At the 150 microns level, however, there was a slight increase in cement dissolution which was statistically significant. The Phase 2 Dynamic study demonstrated that as the marginal gap increased there was a gradual increase in cement dissolution. This increase, however, was not statistically significant. At the 150 micron level there was a statistically significant increase in cement dissolution.

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PII Redacted

VITA

Michael Steven Jacobs [redacted]; the son of Charles Edward Jacobs and Mary Jane Jacobs. He graduated from the University of Houston in June 1973 with a Bachelor of Science in Biology; and, entered the University of Texas Dental School at Houston in July of 1973. At that time he received an Air Force Health Professional Scholarship and was commissioned as a Second Lieutenant. He received his Doctor of Dental Surgery degree in May of 1977.

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In July of 1985 he entered the Post-Doctoral Prosthodontics Program at the University of Texas Health Science Center at San Antonio; and, the following year was admitted as a candidate for the Master of Science degree at the Graduate School of Biomedical Sciences. He was awarded first place in The American College of Prosthodontists First Annual Table Clinic Competition held in San Diego in October of 1987. He has been assigned as a Staff Prosthodontist at Sheppard, Air Force Base, Wichita Falls,Texas.

This thesis is submitted for partial fulfilment of a Master of Science degree to be awarded upon completion of requirements, May 1988, from the University of Texas Health Science Center at San Antonio School of Biomedical Sciences.